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1. Document ID: US 6482325 B1

L2: Entry 1 of 19

File: USPT

Nov 19, 2002

US-PAT-NO: 6482325

DOCUMENT-IDENTIFIER: US 6482325 B1

** See image for Certificate of Correction **

TITLE: Apparatus and process for separation and recovery of liquid and slurry

abrasives used for polishing

DATE-ISSUED: November 19, 2002

INVENTOR-INFORMATION:

NAME

CITY

STATE ZIP CODE

COUNTRY

Corlett; Gary L.

Hollister

CA

Ferri, Jr.; Edward T.

Gilroy

CA

Geatz; J. Tobin

Wilmington NC

US-CL-CURRENT: 210/662; 134/10, 137/93, 210/739, 210/742, 210/743, 210/805, 451/60, 451/88

Full Title Citation Front Review Classification Date Reference Sequences Attachments Claims KMC | Draw Desc Image

2. Document ID: US 6428705 B1

L2: Entry 2 of 19

File: USPT

Aug 6, 2002

US-PAT-NO: 6428705

DOCUMENT-IDENTIFIER: US 6428705 B1

TITLE: Process and apparatus for high flow and low pressure impurity removal

DATE-ISSUED: August 6, 2002

INVENTOR - INFORMATION:

NAME

CITY

STATE

ZIP CODE

COUNTRY

Allen; Stephen D.

Boise

ID

Golden; Josh H.

Santa Cruz

CA

US-CL-CURRENT: 210/638; 210/639, 210/651, 210/652, 210/728, 210/734, 210/735

Full Title Citation Front Review Classification Date Reference Sequences Attachments Claims KWC Draw Desc Image

3. Document ID: US 6379546 B1

L2: Entry 3 of 19

File: USPT

Apr 30, 2002

US-PAT-NO: 6379546

DOCUMENT-IDENTIFIER: US 6379546 B1

TITLE: Method and device for sewage treatment

DATE-ISSUED: April 30, 2002

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Braun; Ulrich 81634 Munchen DE

US-CL-CURRENT: 210/607; 210/167, 210/195.2, 210/196, 210/620, 210/631, 210/650

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | Draw Desc | Image |

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4. Document ID: US 6379538 B1

L2: Entry 4 of 19

File: USPT

Apr 30, 2002

US-PAT-NO: 6379538

DOCUMENT-IDENTIFIER: US 6379538 B1

** See image for Certificate of Correction **

TİTLE: Apparatus for separation and recovery of liquid and slurry abrasives used for polishing

DATE-ISSUED: April 30, 2002

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Corlett; Gary L. Hollister CA
Ferri, Jr.; Edward T. Gilroy CA
Geatz; J. Tobin Wilmington NC

US-CL-CURRENT: 210/96.1; 134/109, 134/902, 210/418

Full Title Citation Front Review Classification Date Reference Sequences Attachments

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KAMC

5. Document ID: US 6361695 B1

L2: Entry 5 of 19 File: USPT Mar 26, 2002

US-PAT-NO: 6361695

DOCUMENT-IDENTIFIER: US 6361695 B1

TITLE: Shipboard wastewater treatment system

DATE-ISSUED: March 26, 2002

Co.

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Husain; HidayatBramptonCABehmann; HenryPuslinchCATonelli; Fernando A.DundasCALiu; Ming GangBramptonCA

US-CL-CURRENT: 210/614; 210/195.2, 210/202, 210/220, 210/259, 210/359, 210/620, 210/621, 210/639

Full Title Citation Front Review Classification Date Reference Sequences Attachments

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6. Document ID: US 6327731 B1

L2: Entry 6 of 19 File: USPT Dec 11, 2001

US-PAT-NO: 6327731

DOCUMENT-IDENTIFIER: US 6327731 B1

TITLE: Clothes washer and dryer system for recycling and reusing graywater

DATE-ISSUED: December 11, 2001

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Back; Dwight D. Melbourne FL Scaringe; Robert P. Rockledge FL Cole; Gregory S. Ormond Beach FL

US-CL-CURRENT: <u>8/158</u>; <u>68/18F</u>, <u>68/207</u>, <u>68/902</u>, <u>8/159</u>

Full Title Citation Front Review Classification Date Reference Sequences Attachments

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7. Document ID: US 6269667 B1

L2: Entry 7 of 19 File: USPT Aug 7, 2001

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US-PAT-NO: 6269667

DOCUMENT-IDENTIFIER: US 6269667 B1

TITLE: Clothes washer and dryer system for recycling and reusing gray water

DATE-ISSUED: August 7, 2001

INVENTOR - INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Back; Dwight D. Melbourne FL Scaringe; Robert P. Rockledge FL Cole; Gregory S. Ormond Beach FL US-CL-CURRENT: 68/17R; 68/18F, 68/20, 68/207, 68/902

8. Document ID: US 6096185 A

L2: Entry 8 of 19

File: USPT

Aug 1, 2000

US-PAT-NO: 6096185

DOCUMENT-IDENTIFIER: US 6096185 A

TITLE: Method and apparatus for recovery of water and slurry abrasives used for chemical and mechanical planarization

DATE-ISSUED: August 1, 2000

INVENTOR - INFORMATION:

NAME

CITY

STATE ZIP CODE

COUNTRY

Corlett; Gary L.

Hollister

CA

Roberson, Jr.; Glenn A.

Carmel

CA

US-CL-CURRENT: 205/742; 204/515, 204/571, 210/175, 210/737, 210/774, 210/775

Full Title Citation Front Review Classification Date Reference Sequences Attachments Draw Desc Image

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KOMC

9. Document ID: US 6071356 A

L2: Entry 9 of 19

File: USPT

Jun 6, 2000

US-PAT-NO: 6071356

DOCUMENT-IDENTIFIER: US 6071356 A

TITLE: Cleaning-in-place with a solution containing a protease and a lipase

DATE-ISSUED: June 6, 2000

INVENTOR-INFORMATION:

NAME

CITY

STATE

ZIP CODE

COUNTRY

Olsen; Hans Sejr

Holte

DK

US-CL-CURRENT: 134/26; 134/27, 134/28, 134/29, 510/111, 510/218, 510/234, 510/392, 510/530

Full Title Citation Front Review Classification Date Reference Sequences Attachments

KANC

10. Document ID: US 6059712 A

L2: Entry 10 of 19

Drawi Desc Image

File: USPT

May 9, 2000

US-PAT-NO: 6059712

DOCUMENT-IDENTIFIER: US 6059712 A

TITLE: Apparatus for continuous separation of fine solid particles from a liquid by

centrifugal force

DATE-ISSUED: May 9, 2000

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Corlett; Gary L. Hollister CA Roberson, Jr.; Glenn A. Carmel CA

US-CL-CURRENT: 494/33; 494/56, 494/67

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<u>L2</u>	L1 and screen	84	<u>L2</u>
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L3: Entry 7 of 7

File: USPT

Sep 25, 1984

DOCUMENT-IDENTIFIER: US 4473474 A TITLE: Charge modified microporous membrane, process for charge modifying said membrane and process for filtration of fluid

Abstract Text (1):
A cationic charge modified microporous membrane, process for producing said membrane and use thereof are provided. The membrane comprises a hydrophilic organic polymeric microporous membrane and a charge modifying amount of a primary cationic charge modifying agent bonded to substantially all of the wetted surfaces of the membrane. The primary charge modifying agent if a water soluble organic polymer having a molecular weight greater than about 1000 wherein each monomer thereof has at least one epoxide group capable of bonding to the surface of the membrane and at least one tertiary amine or quaternary ammonium group. Preferably, a portion of the epoxy groups on the organic polymer are bonded to a secondary charge modifying agent selected from the group consisting of:

Abstract Text (4):
The charge modified microporous membrane of this invention may be used for the filtration of fluids, particularly parenteral or biological liquids. The membrane has low extractables and is sanitizable or sterilizable.

Parent Case Text (3):
This application is also related to copending application U.S. Ser. No. 268,543 filed May 29, 1981 entitled CHARGE MODIFIED MICROPOROUS MEMBRANE, PROCESS FOR CHARGE MODIFYING SAID MEMBRANE, AND PROCESS FOR FILTRATION OF FLUID to Barnes, Jr. et al. This copending application and the content thereof is not prior art with respect to the invention described and claimed in this application.

<u>Brief Summary Text</u> (3):

This invention relates to microporous <u>membranes</u>, and more particularly to cationic charge modified microporous <u>membranes</u> suitable for the filtration of aqueous fluids, such as biological liquids.

Brief Summary Text (5):

Microporous membranes are well known in the art. For example, U.S. Pat. No. 3,876,738 to Marinaccio et al (1975) describes a process for preparing a microporous membrane, for example, by quenching a solution of a film forming polymer in a non-solvent system for the polymer. European patent application No. 0 005 536 to Pall (1979) describes a similar process.

Brief Summary Text (6):
Other processes for producing microporous membranes are described, for example, in the following U.S. Patents:

Brief Summary Text (11):
Commercially available microporous membranes, for example, made of nylon, are available from Pall Corporation, Glen Cove, New York under the trademark ULTIPOR N.sub.66.
Another commercially significant membrane made of polyvinylidene fluoride is available from Millipore Corp., Bedford, Massachusetts under the trademark Durapore. This membrane is probably produced by the aforementioned Grandine, II patents. Such membranes are advertised as useful for the sterile filtration of pharmaceuticals, e.g. removal of microorganisms.

Brief Summary Text (12):

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Various studies in recent years, in particular Wallhausser, Journal of Parenteral Drug Association, June, 1979, Vol. 33, #3, pp. 156-170, and Howard et al, Journal of the Parenteral Drug Association, March-April, 1980, Volume 34, #2 pp. 94-102, have reported the phenomena of bacterial break-through in filtration media, in spite of the fact that the media had a low micrometer rating. For example, commercially available membrane filters for bacterial removal are typically rated as having an effective micrometer rating for the microreticulate membranes structure of 0.2 micrometers or less, yet such membrane typically have only a 0.357 effective micrometer rating for spherical contaminant particles, even when rated as absolute for Ps. diminuta, the conventional test for bacterial retention. Thus passage of a few microorganisms through the membrane may be expected under certain conditions and within certain limits. This problem has been rendered more severe as the medical uses of fifter membranes increases.

Brief Summary Text (14):
One method of resolving this problem and its inevitable consequences, is to prepare a tighter filter, i.e. one with a sufficiently small effective pore dimension to enable the capture of microorganisms, bacterial pyrogen and viral contaminants, by mechanical sieving. Such filter structures, in the form of microporous membranes of 0.1 micrometer rating or less, may be readily prepared. The flow rates, however, exhibited by such structures at conventional pressure drops are prohibitively low. Generally, increasing the pressure drop to provide the desired flow rate is not feasible, even with costly replacement or modification of existing pumping equipment, due to the fact that pressure drop is an inverse function of the fourth power of pore diameter. Thus such modification of the internal geometry, i.e. pore size, of the microporous $\underline{\mathsf{membrane}}$ is not an economical solution to the problem of bacterial breakthrough.

Brief Summary Text (15):

It has long been recognized that adsorptive effects can enhance the capture of particulate contaminants. For example, Wenk in his article "Electrokinetic and Chemical Aspects of Water Filtration", Filtration and Seperation, May/June 1974, indicates that surfactants, PH, and ionic strength may be used in various ways to improve the efficiency of a filter by modifying the charge characteristics of either the suspension, filter or both.

Brief Summary Text (16):

It has also been suggested that adsorptive sequestration, i.e. adsorptive capture of particles by entry into and capture within the pore channels, may in some cases be more important to so-called sterile filtration than bubble point characterization of internal geometry (representing the "largest pore"). Tanney et al, Journal of the Parenteral Drug Association, November-December 1978, Vol. 21, #6 pp. 258-267 demonstrated that adsorptive effects dominate the filtration of flu vaccine through membranes of mixed cellulose esters, cellulose triacetate, and vinyl chlorideacrylonitrile copolymer, counter to the then common understanding of filtration as involving particulate removal by sieve-retention. This is consistent with the observation that bacterial pyrogen and virus particles may be removed by filtration through a membrane even though they are smaller than the pore sizes of commonly used 0.22 micrometer filters. Tanney et al, Journal of the Parenteral Drug Association, January-February, 1979, Vol. 33, #1, pp. 40-51.

Brief Summary Text (18):

Pall et al, Colloids and Surfaces 1 (1980), pp. 235-256, indicates that if the zeta potential of the pore walls of a membrane, e.g. nylon 66, and of the particles are both low, or if they are oppositely charged, the particle will tend to adhere to the pore walls, and the result will be removal of particles smaller than the pores of the filter. Pall et al, however, suggests the use of membranes of substantially smaller pore size to increase the probability of obtaining microbial sterility in filtering fluids.

Brief Summary Text (19):

Zierdt, Applied and Environmental Microbiology, Dec. 1979, pp. 1166-1172, found a strong adherence by bacteria, yeast, erythrocytes, leukocytes, platelets, spores, and polystyrene spheres to membrane materials during filtration through membranes with pore-size diameters much larger than the particles themselves. Zierdt attributed this phenomena to electrostatic forces. The phenomena was partially blocked by pre-treating the filter membrane with a nonionic surfactant. Zierdt found that cellulose membranes adsorbed more bacteria, blood cells and other particles then did polycarbonate filters. Of lesser adsorptive capacity were vinyl acetate, nylon, acrylic, and Teflon membranes. Zierdt additionally found that solvent cast membrane filter materials, e.g. nylon had strong surface charges, whereas ordinary fibrous cellulose materials which are not

solvent cast do not. Zierdt suggested that the development and manufacture of special purpose filter materials with more intrinsic charge than those currently available would extend the usefulness of this phenomenon. Conversely, manufacturing techniques could be developed that would build less intrinsic charge into filters when adsorption is not desired.

Brief Summary Text (20):
Attempts to increase the short life of filter media due to pore blockage and enhance flow rates through filter media having small pores have been made by charge modifying the media by various means to enhance capture potential of the filter. For example, U.S. Pat. Nos. 4,007,113 and 4,007,114 to Ostreicher, describe the use of a melamine formaldehyde cationic colloid to charge modify fibrous and particulate filter elements; U.S. Ser. No. 147,975 filed May 8, 1980, now U.S. Pat. No. 4,305,782, to Ostreicher et al describes the use of an inorganic cationic colloidal silica to charge modify such elements; and copending U.S. Ser. No. 164,797 filed June 30, 1980, to Ostreicher et al, describes the use of a polyamido-polyamine epichlorhydrin cationic resin to charge modify such filter elements. None of these references teach or suggest charge modifying an organic polymeric microporous membrane, nor do any of the filtration media described therein, e.g. fiber and/or particulate, provide the advantages of such a membrane.

Brief Summary Text (23):
There are numerous references which describe the treatment of porous membranes for various objects. U.S. Pat. No. 3,556,305 to Shorr (1971) describes a tripartite membrane for use in reverse osmosis. The membrane comprises an anisoptropic porous substrate, an ultra-thin adhesive layer over the porous substrate, and a thin diffusive membrane formed over the adhesive layer and bound to the substrate by the adhesive layer. The anisotropic porous membranes used in Shorr are distinguished from isotropic, homogeneous membrane structures whose flow and retention properties are independent of flow direction. Such isotropic membranes do not function properly when utilized in the invention of Shorr.

Brief Summary Text (24): $\overline{\text{U.S. Pat. No. 3,556,992}}$ to Massuco (1971) describes another anisotropic ultra-filtration membrane having thereon an adhering coating of irreversibly compressed gell.

Brief Summary Text (25):
U.S. Pat. No. 3,808,305 to Gregor (1974) describes a charged membrane of macroscopic homogeneity prepared by providing a solution containing a matrix polymer, polyelectrolytes (for charge) and a cross-linking agent. The solvent is evaporated from a cast film which is then chemically cross-linked. The membranes are used for ultrafiltration.

Brief Summary Text (26):
U.S. Pat. Nos. 3,944,485 (1976) and 4,045,352 (1977) to Rembaum et al describe ion exchange hollow fibers produced by introducing into the wall of the pre-formed fiber, polymerizable liquid monomers. The monemers are then polymerized to form solid, insoluble, ion exchange resin particles embedded within the wall of the fiber. The treated fibers are useful as membranes in water-treatment, dialysis, and generally to separate ionic solutions. U.S. Pat. No. 4,014,798 to Rembaum (1977) describes similar type hollow fiber using different type resins to produce the ion exchange mechanism.

Brief Summary Text (27):

U.S. Pat. No. 4,005,012 to Wrasidlo (1977) describes a process for producing a semi-permeable anisotropic membrane useful in reverse osmosis processes. The membranes are prepared by forming a polymeric ultra-thin film, possessing semi-permeable properties on a microporous support. Such an ultra-thin film may be formed by contacting an amine modified polyepihalohydrin with a polyfunctional agent and depositing this film on the external surface of a microporous substrate. Preferred semipermeable membranes are polysulfone, polystyrene, cellulose butyrate, cellulose nitrate and cellulose acetate.

Brief Summary Text (28): $\overline{U.S.}$ Pat. No. 4,125,462 to Latty (1978) describes a coated semi-permeable reverse osmosis membrane having an external layer or coating of a cationic polyelectrolyte preferably poly(vinylimidazoline) in the bi-sulfate form.

Brief Summary Text (29): U.S. Pat. No. 4,214,020 to Ward et al (1980) describes a novel method for coating the

exteriors of a bundle of hollow-fiber semipermeable membranes for use in fluid separations. Typical polymers coated are polysulfones, polystyrenes, polycarbonates, cellulosic polymers, polyamides and polyimides. Numerous depositable materials are listed, see col. 10, lines 55-col. 12, for example, poly(epichlorhydrin) or polyamides.

Brief Summary Text (30):

U.S. Pat. No. 4,239,714 to Sparks et al (1980) describes a method of modifying the pore size distribution of a microporous separation media so as to provide it with a sharp upper cut-off of a pre-selected molecular size. This is accomplished by effectively blocking the entrances to all of the pores of the separation media larger than a pre-selected molecular size constituting the desired cut-off, but leaving unchanged the smaller pores. The separation media may be in the form of polymeric membranes, e.g. cellulose acetate, cellulose nitrate, polycarbonates, polyolefins, polyacrylics, and polysulfones. The foregoing is accomplished by filling the pores of the membrane with a volatile liquid and then evaporating the liquid to form voids at the entrances to the pores. A concentrated solution of a cross-linkable or polymerizable pore blocking agent, such as protein, enzyme, or polymeric maerials is then applied to the surface of the membrane.

Brief Summary Text (31):

U.S. Pat. No. 4,250,029 to Kiser et al (1981) describes coated membranes having two or more external coatings of polyelectrolytes with at least one oppositely charged adjacent pair separated by a layer of material which is substantially charge neutralized. Kiser et al is primarily directed to the use of charged membranes to repel ions and thereby prevent passage through the membrane pores. The coated membranes are described as ordinary semi-permeable membranes used for ultrafiltration, reverse osmosis, electrodialysis or other filtration processes. A microscopic observation of the coated membranes shows microscopic hills and valleys of polyelectrolyte coating formed on the original external smooth skin of the membrane. The membranes are particularly useful for deionizing aqueous solutions. Preferred membranes are organic polymeric membranes used for ultra-filtration and reverse osmosis processes, e.g., polyimide, polysulfone, aliphatic and aromatic nylons, polyamides, etc. Preferred membranes are anisotropic hollow fiber membranes having an apparent pore diameter of from about 21 to about 480 angstroms.

Brief Summary Text (32):
Charge modified membranes are disclosed in U.S. Ser. No. 358,822 of Ostreicher filed May 9, 1973, now abandoned (corresponding to Japanese Pat. No. 923649 and French Pat. No. 7415733). As disclosed therein, an isotropic cellulose mixed ester membrane, was treated with a cationic colloidal melamineformaldehyde resin to provide charge functionality. The membrane achieved only marginal charge modification. Additionally, the membrane was discolored and embrittled by the treatment, extractables exceeded desirable limits for certain critical applications, and the membrane was not thermally sanitizable or sterilizable. Ostreicher also suggests such treatment for the nylon membranes prepared by the methods described in U.S. Pat. No. 2,783,894 to Lovell (1957) and U.S. Pat. No. 3,408,315 to Paine (1968). It has been demonstrated that nylon microporous membranes treated according to Ostreicher would also demonstrate marginal charge modification, high extractables and/or inability to be thermally sanitizable or sterilizable.

Brief Summary Text (34): U.S. Pat. No. 3,497,451 to Hoehn et al (1970)--the use of "type 8" nylon for the desalination of sea $\underline{\text{water}}$;

Brief Summary Text (35):
U.S. Pat. No. 3,615,024 to Michaels (1968) -- an anisotropic reverse osmosis membrane which may be nylon;

Brief Summary Text (36): U.S. Pat. No. 4,148,606 to Morita et al $(1979)^{\frac{1}{2}}$ -a method of sterilizing a dialyzer by irradiating the semipermeable membrane in the presence of an antibacterial agent; and

Brief Summary Text (39): It is an object of this invention to provide a novel cationic charge modified microporous membrane, particularly suitable for the filtration of biological or parenteral liquids.

Brief Summary Text (40):

It is a further object of this invention to provide a process for cationically charge modifying a hydrophilic organic polymeric microporous membrane.

Brief Summary Text (42):

It is another object of this invention to provide an isotropic cationic charge modified microporous membrane which has low extractables suitable for the filtration of biological or parenteral liquids.

Brief Summary Text (43):

It is yet another object of this invention to prepare a sanitizable or sterilizable microporous membrane for the efficient removal of bacteria, viruses and pyrogen from contaminated liquids.

Brief Summary Text (44):

A still further object of this invention is to enhance the adsorptive sequestration capacity of microporous membranes without affecting the internal microreticulate structure.

Brief Summary Text (45):

It is a further object of this invention to enhance the capture potential of a preformed microporous membrane without decreasing the pore size of the membrane.

Brief Summary Text (46):

It is still a further object of this invention to provide a microporous membrane capable of capturing anionic particulate contaminant of a size smaller than the effective pore size of the membrane.

Brief Summary Text (47):

These and other objects of this invention are attained by a novel cationic charge modified microporous membrane. The membrane comprises a hydrophilic organic polymeric microporous membrane and a charge modifying amount of a primary cationic charge modifying agent bonded to substantially all of the wetted surfaces of the membrane, the primary charge modifying agent being a water soluble organic polymer having a molecular weight greater than about 1000 wherein each monomer thereof has at least one epoxide group capable of bonding to the surface of the membrane and at least one tertiary amine or quaternary ammonium group.

Brief Summary Text (51):

The invention is further directed to a process for cationically charge modifying a hydrophilic organic polymeric microporous membrane by applying to the membrane the aforesaid charge modifying agents. Preferably, the process for charge modifying the microporous membrane comprises contacting the membrane with aqueous solutions of the charge modifying agents.

Brief Summary Text (52):

The preferred microporous membrane is nylon, the preferred primary and secondary charge modifying agents are, respectively, polyamido-polyamine epichlorohydrin and tetraethylene pentamine.

Brief Stomary Text (53):
The invention is further directed to a process for improving the responsiveness of a nylon membrane to charge modification, comprising chemically treating the membrane to provide enhanced ninhydrin response evidencing free amino functionality.

Brief Summary Text (54):

The charge modified microporous membrane of this invention may be used for the filtration of fluids, particularly parenteral or biological liquids.

Drawing Description Text (2):

FIG. 1 is a graphical representation showing performance data, i.e. filtration efficiency, for a 0.2 micrometer membrane treated in accordance with the invention as compared to an untreated membrane, both challenged with 0.109 contaminant at 0.5 gpm/ft..sup.2 (0.002 lpm/cm.sup.2) (See Example VII.)

Drawing Description Text (3):

FIGS. 2 and 3 are Scanning Electron Microscope (SEM) photos of the cationic charge modified membrane of this invention challenged with contaminant (See Example VII.)

Detailed Description Text (2):

The cationic charge modified microporous membrane of this invention is produced from a hydrophilic organic polymeric microporous membrane. Such membranes are well known in the art.

Detailed Description Text (3):

By the use of the term "microporous membrane" as used herein, it is meant a substantially symmetrical, isotropic porous membrane having a pore size of at least 0.05 microns or larger or an initial bubble point (IBP), as that term is used herein, in water of less than 120 psi. A maximum pore size useful for this invention is about 1.2 micron or an IBP of greater than about 10 psi. By "symmetrical" it is meant that the pore structure is substantially the same or both sides of the membrane. A number of commercially available membranes not encompassed by the term "microporous membrane" are "asymmetric", i.e. having one side formed with a very light thin layer which is supported by a much more porous open structure. By the use of the term "isotropic", it is meant the membrane has a uniform pore structure throughout the membrane.

Detailed Description Text (4):

By the use of the term "hydrophilic" in describing the microporous membrane, it is meant a membrane which adsorbs or absorbs water. Generally, such hydrophilicity is produced by a sufficient amount of hydroxyl (OH--), carboxyl (--COOH) amino (--NH.sub.2) and/or similar functional groups on the surface of the membrane. Such groups assist in the adsorption and/or absorption of the water onto the membrane. Such hydrophilicity of the membrane is a necessary element of this invention to provide the adequate bonding of the primary charge modifying agent through the epoxide substituent to the microporous membrane.

Detailed Description Text (5):

A preferred microporous membrane is one produced from nylon. The term "nylon" is intended to embrace film forming polyamide resins including copolymers and terpolymers which include the recurring amido grouping.

Detailed Description Text (10):

The preferred membrane substrates are produced by the method disclosed in U.S. Pat. No. 3,876,738 to Marinaccio et al. Another method of producing such membranes is described in European patent application No. 0 005 536 to Pall. The entire disclosures of both of these references are incorporated herein by reference.

Detailed Description Text (11):

Additionally, any of the hydrophilic microporous membranes commercially available, for example, Pall Corp.'s ULTIPOR N.sub.66 (nylon), Millipore's Durapore (polyvinylidene fluoride), and cellulose acetate/nitrate membranes produced by numerous companies, having characteristics potentially suitable for fine filtration of fluids, particularly aqueous systems, are suitable for treatment in accordance with this invention.

Detailed Description Text (12):

The Marinacio et al process for producing membrane develops a unique fine internal microstructure through the quench technique described therein, offering a superior substrate for filtration. Broadly, Marinaccio et al produces microporous films by casting or extruding a solution of a film-forming polymer in a solvent system into a quenching bath comprised of a non-solvent system for the polymer. Although the non-solvent system may comprise only a non-solvent, the solvent system may consist of any combination of materials provided the resultant non-solvent system is capable of setting a film and is not deleterious to the formed film. For example, the non-solvent system may consist of materials such as water/salt, alcohol/salt or other solvent-chemical mixtures. The Marinaccio et al process is especially effective for producing nylon films. More specifically, the general steps of the process involve first forming a solution of the film-forming polymer, casting the solution to form a film and quenching the film in a bath which includes a non-solvent for the polymer.

Detailed Description Text (15):

The nylon solutions after formation are diluted with non-solvent for nylon and the non-solvent employed is miscible with the nylon solution. Dilution with non-solvent may, according to Marinaccio et al, be effected up to the point of incipient precipitation of the nylon. The non-solvents are selected on the basis of the nylon solvent utilized. For example, when water-miscible nylon solvents are employed, water can be employed. Generally, the non-solvent can be methyl formate, aqueous lower alcohols, such as methanol and ethanol, polyols such as glycerol, glycols, polyglycols, and ethers and esters thereof, water and mixtures of such compounds. Moreover, salts can also be used to control solution properties.

Detailed Description Text (16):

The quenching bath may or may not be comprised of the same non-solvent selected for preparation of the nylon solution and may also contain small amounts of the solvent employed in the nylon solution. However, the ratio of solvent to non-solvent is lower in the quenching bath than in the polymer solution in order that the desired result be obtained. The quenching bath may also include other non-solvents, e.g., water.

Detailed Description Text (20):

Pall's aforementioned European patent application No. 0 005 536 describes another similar method for the conversion of polymer into microporous membrane which may be used. Broadly, Pall provides a process for preparing skinless hydrophilic alcohol-insoluble polyamide membranes by preparing a solution of an alcohol-insoluble polyamide resin in a polyamide solvent. Nucleation of the solution is induced by the controlled addition to the solution of a nonsolvent for the polyamide resin, under controlled conditions of concentration, temperature, addition rate, and degree of agitation to obtain a visible precipitate of polyamide resin particles (which may or may not partially or completely redissolve) thereby forming a casting solution.

Detailed Description Text (21):

The casting solution is then spread on a substrate to form a thin film. The film is then contacted and diluted with a mixture of solvent and nonsolvent liquids containing a substantial proportion of the solvent liquid, but less than the proportion in the casting solution, thereby precipitating polyamide resin from the casting solution in the form of a thin skinless hydrophilic membrane. The resulting membrane is then washed and drivi.

Detailed Description Text (22):

In Pall's preferred embodiment of the process, the solvent for the polyamide resin solution is formic acid and the nonsolvent is <u>water</u>. The polyamide resin solution film is contacted with the nonsolvent by immersing the film, carried on the substrate, in a bath of nonsolvent comprising water containing a substantial proportion of formic acid.

Detailed Description Text (23):

These preferred nylon membranes, i.e. described in Marinaccio et al and Pall, are characterized by an isotropic structure, having a high effective surface area and a fine internal microstructure of controlled pore dimensions with narrow pore size distribution and adequate pore volume. For example, a representative 0.22 micrometer rated nylon 66 membrane (polyhexamethylene adipamide) exhibits an initial bubble point (IBP) of about 45 to 50 psid., a foam all over point (FAOP) of about 50 to 55 psid, provides a flow of from 70 to 80 ml/min of water at 5 psid (47 mm. diameter discs), has a surface area (BET, nitrogen adsorption) of about 13 m.sup.2 /g and a thickness of about 4.5 to 4.75 mils.

Detailed Description Text (24):

The primary charge modifying agent used in this invention is a water soluble organic polymer having a molecular weight greater than about 1000, wherein the monomer has at least one epoxide substituent capable of bonding to the surface of the membrane and at least one tertiary amine or quaternary ammonium group capable of providing a cationic charge site.

Detailed Description Text (25):

The primary charge modifying agent is bonded to substantially all of the wetted surface of the microporous membrane. By the use of the term "bonded" it is meant that the charge modifying agent(s) are sufficiently attached to the membrane and/or to each other so that they will not significantly extract under the intended conditions of use. By the use of the term "substantially all of the wetted surface" as used herein it is meant all of the external surface and internal pore surfaces which are wetted by a fluid passing through the membrane or in which the membrane is immersed.

Detailed Description Text (36):

The polyamide may be derived form the reaction of a polyalkylene polyamine and a saturated aliphatic dibasic carboxyic acid containing from about 3 to 10 carbon atoms. The polyamide produced is water soluble and contains the recurring groups:

Detailed Description Text (37):

where n and x are each 2 or more and R is the divalent hydrocarbon radical of the dicarboxylic acid. This polyamide is then reacted with epichlorohydrin to form the preferred water soluble charge modifiers used in this invention.

Detailed Description Text (60):

The secondary charge modifying agent is bonded to the microporous membrane by bonding to a portion of the epoxide substituents of the polymeric primary charge modifying agent.

Detailed Description Text (61):

The amount of primary and secondary cationic charge modifying agent utilized is an amount sufficient to enhance the electropositive capture potential of the microporous membrane. Such an amount is highly dependent on the specific charge modifying agents utilized.

Detailed Description Text (62):

Broadly, the process of this invention is directed to cationically charge modifying a hydrophilic organic polymeric microporous membrane, e.g. nylon. The process comprises applying to the membrane a charge modifying amount of the primary cationic charge modifying agent bonded to the membrane structure through the epoxide substituent. Preferably, the process comprises (a) contacting the membrane with an aqueous solution of the primary cationic charge modifying agent and (b) contacting the membrane with an aqueous solution of the secondary charge modifying agent. The contacting steps may be performed in any order, i.e. step (a) prior to step (b) or vice versa. It is preferred, however, for optimum (minimum) extractables to first contact the membrane with an aqueous solution of the primary cationic charge modifying agent and then subsequently contact the so treated membrane with the aqueous solution of the secondary charge modifying agent.

Detailed Description Text (63):

In order to provide the charge modifying amount of cationic charge modifying agent to the membrane, it is preferred that the aqueous solution of primary charge modifying agent that the membrane is contacted with contain at least about 1.0% charge modifying agent, by weight of the solution. The maximum amount of charge modifying agent in the aqueous solution is limited by economic and solubility limitations. For example, an excess of primary charge modifying agent which is not bonded to the microporous membrane will not be economically utilized and will constitute an undesirable extractive from the membrane. It has been found that the amount of charge modifying agent in the aqueous solution should probably not exceed about 5% by weight of the solution.

Detailed Description Text (64):

The amount of secondary charge modifying agent used in the aqueous solution is highly dependent on the specific secondary charge modifying agent and the amount and type primary charge modifying agent used, and the cross-linking mechanism between these compounds to provide the bonding of such charge modifying agent to the microporous membrane. For general guidance however, it has been found that a weight ratio of primary to secondary charge modifying agent of from about 2:1 to about 500:1, preferably from about 25:1 to about 75:1 in the aqueous solutions contacted with the membrane, is generally sufficient to provide the bonding of the cationic charge modifying agents to the membrane. It has been found that if the aqueous solution containing the secondary charge modifying agent contains at least about 0.01% charge modifying agent by weight of the solution, up to a maximum of about 0.5% weight of the solution when used in conjunction with the aforementioned aqueous solution of primary charge modifying agent, that adequate bonding of the charge modifying agents to the microporous membrane is obtained.

Detailed Description Text (65):

Both the charge modifying agents may be contacted with the membrane by dipping the membrane in the aqueous solutions of these compounds for a period of time sufficient to effect the desired degree of pick-up. Alternatively, the charge modifying agents may be applied by spraying or contacting a wick or roll along the surface of the microporous membrane.

Detailed Description Text (66):

Preferably, the hydrophilic membranes, in particular the nylon membrane produced according to the aforementioned Marinaccio et al, are surface activated, as with a caustic or basic wash, i.e. above pH 7, to provide a surface affording an enhanced amino-functionality as measured, for example, by ninhydrin colorimetery. This provides improved responsiveness to charge modification.

Detailed Description Text (67):

In an embodiment of this invention the membrane is surface activated before contacting charge modifying agents with the membrane. In the simplest form, the preferred nylon membrane is treated with caustic to provide additional amino groups for cross-linking. The same or enhanced effect is preferably achieved by providing an alkaline bath for application of at least one of the charge modifying agents, preferably the primary charge modifying agents. The bath is preferably at a pH of about 9 to about 11.

Detailed Description Text (68):

It is believed that the foregoing caustic surface treatment provides surface functionality free of blocking salts which are produced, for example, by the reaction of the acids used in producing the hydrophilic membrane (see, for example, Marinaccio et al and Pall) with the functional substituents, e.g. amino. Thus the caustic treated nylon membrane evidences an enhanced ninhydrin test response for free amino functionality.

Detailed Description Text (73):

Although applicants do not wish to be bound by the following theory, it is believed that in bonding the primary charge modifying agent to the microporous membrane the epoxide groups on the primary charge modifying agent enter into addition type reactions with the hydroxyl, carboxyl and primary and secondary amines, which are on the hydrophilic microporous membrane and the primary and secondary cationic charge modifying agents. These reactions may be represented as follows: ##STR8##

Detailed Description Text (75):

1. The epoxide cross-links the primary amine groups on the hydrophilic membrane to the primary charge modifying agent and the primary and/or secondary amine groups on the secondary charge modifying agent;

Detailed Description Text (76):

2. The epoxide cross-links the carboxyl groups on the hydrophilic microporous membrane to the primary charge modifying agent and the primary and/or secondary amine groups of the secondary charge modifying agent;

Detailed Description Text (78):

It is theorized that a polymeric primary charge modifying agent offering greater than three epoxide groups per monomer offers no benefit, and in fact may limit the coupling reactions of the primary charge modifying agent by steric hindrance. Additionally, the presence of unreacted epoxide groups on the charge modified microporous membrane may be undesirable in the finished charge modified membrane.

Detailed Description Text (81):

Amine groups of all three classes, i.e. primary, secondary or tertiary are capable of forming hydrogen bonds with water. As a result; amines of relatively low molecular weight, i.e. short carbon chain length are quite soluble in water, with border line solubility in water occurring at about 6 carbon atoms per amine group. In the preferred embodiment of this invention it is highly desirable that the cationic charge modifying agents be soluble in water to provide the desired environment for production, i.e. elimination of fumes, toxicity, etc.

Detailed Description Text (84):

It is this latter characteristic, that produces an undesirable reduction in positive surface charge on the microporous membrane (as measured by electrophoretic mobility or streaming potential), and the corresponding reduction in adsorptive capacity for anionic contaminants that has been noted when amine charge modified filter media is tested over a series of increasing pHs. It would therefore appear that the more basic the amine charge modifying agent, the higher is the charge modification and adsorptive capacity for contaminants that a filter media, e.g. membrane, will exhibit at a given

Detailed Description Text (85):

Basicity of an amine is defined by measuring the extent to which the amine can accept hydrogen ions from water, with the equilibrium constant for this reaction being the basicity constant Kb: ##EQU1##

Detailed Description Text (87): To select from among the aliphatic amines a preferred embodiment on a theoretical basis becomes somewhat more complicated due to the fact that one is concerned with the basicity of the amine bonded through the epoxide to the microporous membrane.

Detailed Description Text (89):

Preferably, between each contacting step of the process for producing the membrane, the membrane is drained for a period of time sufficient to remove most of the water and chemical compound not absorbed or adsorbed onto the surface of the membrane. Optionally, the membrane may be transferred directly from the first contacting step to the second contacting step, although this is less preferred. The intermediate treatment may also be a restrained drying step.

Detailed Description Text (90):

After ::- microporous membrane has been contacted with the aqueous solution, it is then dried and cured, preferably in a restrained condition to prevent shrinkage.

Detailed Description Text (91):

Drying of the membrane under restraint is described in the Assignee's co-pending U.S. Ser. No. 201,086 to Repetti filed Oct. 27, 1980 now Defensive Publication No. T-103,601. The entire disclosure of this application is incorporated herein by reference. Generally, any suitable restraining technique may be used while drying, such as winding the membrane tightly about a drying surface, e.g. a drum. Biaxial control is preferred and tensioning the membrane on a stretching frame is considered the most preferred. Preferably, the restraint imposed affects no reduction in dimensions.

Detailed Description Text (92):

Final drying and curing temperatures should be to dry and cure the treated membranes, preferably from about 120.degree. C. to 140.degree. C. for minimization of drying times without embrittlement or other detrimental affects to the membrane.

Detailed Description Text (93):

The completed membrane may be rolled and stored for use under ambient conditions. It will be understood that the treated membrane may be supplied in any of the usual commercial forms, for example, as discs or pleated cartridges.

Detailed Description Text (94):

The present invention provides an integral, coherent microporous membrane of retained internal pore geometry. The charge modified membrane has an improved effective filtration rating relative to the untreated micro-reticulate polymer structure. Such improvement is brought about by charge sites or regions which are effective during fltration to enhance filtration performance through eletrokinetic effects.

Detailed Description Text (95):

The resulting membrane offers improved micrometer rating at equivalent flow and capacity with retention of membrane structure, yet without evidence of significant resin extractables. In effect the effective micrometer rating for contaminant particles is less than the effective micrometer rating of the microreticulate membrane structure. By the use of the term "effective micrometer rating for contaminant particles", it is meant the actual size of the particles that the membrane will quantitatively remove from the fluid being filtered. By the use of the term "effective micrometer rating of the microreticulate membrane structure" it is meant the size of the particulate that would pass through the membrane if all adsorptive effects of the membrane were eliminated.

Detailed Description Text (96):

For so-called sterile filtrations involving biological liquids, the filter is sanitized or sterilized by autoclaving or hot water flushing. Accordingly, the charge modified membrane must be resistant to this type treatment, and must retain its integrity in use. Any modification to the filter structure, especially brought about by chemical agents which may be unstable under conditions of treatment and use, must be scrutinized with care to minimize the prospect of extractables contaminating the filtrate, interfering with analyses and potentially introducing harmful toxins to a patient. Specifically, any such filter must meet the test standards in the industry, e.g. ASTM D 3861-79 (incorporated herein by reference), and generally prove less than 5 mg. of extractables in 250 ml solvent (water at 80.degree. C.; 35% ethanol at room temperature) for a 293 mm diameter disc.

Detailed Description Text (97):

While the primary and secondary charge modifying agents afford cross-linking functionality and cross-linking through such functionality with the base membrane, the improved accessibility of the reactive groups on the membrane brought about by the activation treatment, i.e. caustic wash, enhances interreaction of the agents with the membrane, and insures extremely low extraction levels. Where extraction levels are of major concern, this represents the most prefered embodiment.

Detailed Description Text (98):

The resulting charge modified membrane is characterized by retention of internal microstructure, thus offering essentially the same flow characteristics as the untreated membrane. For example, a 0.22 micrometer rated nylon membrane is essentially absolute for 0.109 test beads.

Detailed Description Text (99):

The charge modified membrane additionally is easy to handle and readily formed into convoluted structures, e.g. pleated configurations. By reason of its retained flow characteristics, it may be employed directly in existing installations, without pumping modifications. These favorable properties are secured without sacrifice to other characteristics. The membrane may also be constructed to meet or exceed extractable requirements.

Detailed Description Text (101):

Filter membranes in accordance with this invention may be employed alone or in combination with other filter media to treat pharmaceuticals such as antibiotics, saline, solutions, dextrose solutions, vaccines, blood plasma, serums, sterile water or eye washes; beverages, such as cordials, gin, vodka, beer, scotch, whisky, sweet and dry wines, champagne or brandy; cosmetics such as mouthwash, perfume, shampoo, hair tonic face cream or shaving lotion; food products such as vinegar, vegetable oils; chemicals such as antiseptics, insecticides, photographic solutions, electroplating solutions, cleaning compounds, solvent purification and lubrication oils; and the like for retention of submicronic particles, removal of bacterial contaminants and resolution of colloidal hazes. Illustratively, in hospital usage, membrane filters are employed to concentrate abnormal exfoliated cells from a vaginal rinse, to isolate blood parasites from peripheral blood, or bacteria from serum or leucocytes and casts from urine.

Detailed Description Text (102):

In the case of preparation for use in sterile filration, the membrane is thermally sanitized or sterilized as by treatment in an autoclave at 121.degree. C. under 15 psig. for 1 hour, or hot water flushing at 85.degree. F. for 1 hour.

Detailed Description Text (107):

The dry membrane thickness was measured with a 1/2 inch (1.27 cm) diameter platen dial indicator thickness gauge. Gauge accuracy was +0.00005 inches (+0.05 mils).

Detailed Description Text (109):

 \overline{A} 47 mm diameter disc of the membrane sample is placed in a special test holder which seals the edge of the disc. Above the membrane and directly in contact with its upper face, is a perforated stainless steel support screen which prevents the membrane from deforming or rupturing when air pressure is applied to its bottom face. Above the membrane and support screen, the holder provides a inch deep cavity into which distilled water is introduced. A regulated air pressure is increased until a first stream of air bubbles is emitted by the <u>water</u> wetted <u>membrane</u> into the quiescent pool of water. The air pressure at which this first stream of air bubbles is emitted is called the Initial Bubble Point (IBP) of the largest pore in that membrane sample--see ASTM D-2499-66T.

Detailed Description Text (110):

Once the Initial Bubble Point pressure has been determined and recorded, the air pressure is further increased until the air flow thru the wetted membrane sample, as measured by a flow meter in the line between the regulator and the sample holder, reaches 100 cc/min. The air pressure at this flow rate, is called the Foam-All-Over-Point (FAOP), and is directly proportional to the mean pore diameter of the sample membrane. In this series of tests, these two parameters (IBP and FAOP) are used to determine if any change has occurred in the maximum or mean pore size of the membrane sample as a result of the charge modifying process utilized.

Detailed Description Text (112):

A 47 mm. diameter disc of the membrane sample is placed in a test housing which allows pressurized water to flow thru the membrane. Prefiltered water is passed thru the membrane sample at a pressure differential of 5 psid. A graduate cylinder is used to measure the volume of water passed by the membrane sample in a one minute period. In this series of tests this parameter is used in conjunction with the IBP and FAOP to

determine if any reduction in pore size or pore blockage has occurred as a result of the charge modifying process utilized.

Detailed Description Text (114):

A 47 mm diameter disc of the membrane sample is placed in a test housing which allows pressurized water flow thru the membrane. The challenge solution consists of distilled water at a pH of 7.0, and Metanil Yellow dye. The dye inlet concentration is adjusted to produce a 76 percent transmittance at a wavelength of 430 nm, as measured on a Perkin Elmer Model 295 Spectrophotometer. By means of a peristaltic pump the challenge solution is flowed thru the membrane sample at a flow rate of 28 ml/min. The transmittance of the effluent is measured by passing it thru a constant flow cell in the aforementioned Spectrophotometer. The effluent transmittance and pressure drop across the membrane is measured and recorded as a function of time. The test is terminated when the effluent transmittance increases to 85 percent of the inlet transmittance. In this series of tests, the length of time that it takes to reach the 85 percent transmittance in the effluent is called the "breakthru" time. Since the Metanil Yellow is a low molecular weight anionic dye incapable of being mechanically removed (filtered) by the membrane, this breakthru time is proportional to the cationic adsorptive capacity of the membrane sample. This test is therefore used to determine the effectiveness of the charge modification technique.

Detailed Description Text (116):

Extractables were determined by ASTM D-3861-79. The quantity of water-soluble extractables present in the membrane filters was determined by immersing the preweighed membrane in boiling reagent grade water for an extended time and then drying and reweighing the membrane. A control membrane was employed to eliminate weighing errors caused by balance changes or changing moisture content of the membrane in the weighing procedures. Weight changes of the control membrane were applied as a correction factor to the weight change of the test membrane filters.

Detailed Description Text (118): PREPARATION OF MICROPOROUS MEMBRANE

Detailed Description Text (119):
A representative nylon 66 membrane of 0.22 micrometer nominal rating, having a nominal surface area of about 13 m.sup.2 /g, an Initial Bubble Point of about 47 psi, a Foam-All-Over-Point of about 52 psi was prepared by the method of Marinaccio et al, U.S. Pat. No. 3,876,738, utilizing a dope composition of 16 percent by weight nylon 66 (Monsanto Vydyne 66B), 7.1% methanol and 76.9% formic acid, a quench bath composition of 25% methanol, 75% water by volume (regenerated as required by the method of Knight et al, U.S. Pat. No. 3,928,517) a casting speed of 24 inches/minute (61 cm/min), and a quench bath temperature of 20.degree. C. The membrane was cast just under the surface of the quench bath by application to a casting drum rotating in the bath (9 to 10 mils as cast wet, to obtain 4.5 to 5.5 mils dry) and allowed to separate from the drum about 90.degree. of arc from the point of application, the self-supporting membrane forming a shallow catenary to takeup. A portion of the uniform opaque film was dried (in restrained condition to resist shrinkage) in a forced air oven at 80.degree.-90.degree. C. for 30 minutes.

Detailed Description Text (122): PREPARATION OF CHARGE MODIFIED MICROPOROUS MEMBRANE

Detailed Description Text (123):

1. Membrane samples (dried and undried) were dipped in a bath of Hercules 1884 polyamido-polyamine epichlorohydrin resin (4% solids by weight), and allowed to attain adsorption equilibrium. The treated membrane samples were washed to remove excess resin and dried in restrained condition on a drum at a temperature of 110.degree. C. for a period of about 3 minutes.

Detailed Description Text (124):

The treated membrane samples were compared for flow and bubble point characteristics as follows, and found to be essentially identical for treated and untreated samples, evidencing retention of pore and surface geometry. The results are set forth in Table

Detailed Description Text (125):

Thus, in terms of the morphological and hydrodynamic parameters that control mechanical sieving, the filtration characteristics of the treated membranes were essentially identical with the untreated nylon membrane.



Detailed Description Text (126):

2. Similar characterizations were conducted on another membrane sample, similarly prepared, but treated with 2% Hercules R4308 resin (a free radical polymerized resin based upon diallyl nitrogen-containing materials, reacted with epichlorohydrin) in a bath adjusted to pH 10.5, overcoated with 0.1% tetraethylene pentamine, dried, cured, washed and redried. The results are set forth in Table II.

Detailed Description Text (127):

Surface area of the treated and untreated membranes remained essentially unchanged; tensile strength increased with treatment with some loss in elongation. The treated sheet was more flexible; creasing of the untreated sheet resulted in cracking and splitting.

Detailed Description Text (130):

The Hercules 1884 treated membrane samples (Example I.B.1.) were subjected to the filtration tests indicated below:

Detailed Description Text (135):

MS-2 bacteriophage was added to Houston, Tex. (U.S.A.) tap water to produce a concentration of 3.4.times.10.sup.5 PFU/ml PFU is "Plaque Forming Unit"), and 10 ml was passed through each of the test filters mounted in a 25 mm diameter stainless steel holder. Effluents were analyzed for viral content by standard techniques. Results are set forth in Table IV:

Detailed Description Text (139):

The test filters were challenged with a solution of blue food coloring dye (FD & C No. 1). The solution had a light transmittance of 62.5% at 628 nm. The light transmittance of the effluent was monitored and removal efficiencies determined (based on distilled water light transmittance=100%). Results are set forth in Table VI.

Detailed Description Text (141):

In a series of related run employing dried Hercules 1884 treated membrane as described in Example I.B. 1. the amount of charge modifying resin in the treatment bath was modified from 1 to 5% by weight, the drying time for the treated membrane was altered from 15 to 30 minutes, the adsorption equilibration time was modified from 1 to 5 minutes, and pH was shifted from 4 to 9 and the factorial experiment analyzed for responses.

Detailed Description Text (145):

Alkaline pH conditions reduced extractables markedly, and the treated membrane showed improved flow rate and filtration at essentially equivalent pressure drop.

Detailed Description Text (147):

A. A non-treated microporous nylon membane prepared in accordance with Example IA was soaked in ninhydrin (1,2,3-trione-2-hydrate, 0.4% aqueous) overnight, and examined for color development. The membrane had turned a light purple, evidencing a good level of amino functionality (positive test ranges from blue to purple). A competitive Pall Corp. nylon membrane (non-charge modified) evidenced only a very pale hint of lavender tint in the same test.

Detailed Description Text (148):

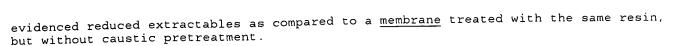
The same membrane of Example IA was washed with a saline solution and soaked in saturated sodium tetraborate. The membrane tested positive for amino-functionality (yellow to orange color) with the addition of 3-4 drops of 2,4,6-trinitro benzene sulfonic acid (3% aqueous). The solution turning a muted yellow within 30 seconds. The competitive Pall membrane showed no color response.

Detailed Description Text (149):

B. The untreated membrane of Example IA was treated with caustic (1 minute immersion in aqueous sodium hydroxide ph 10.5) washed, dried and soaked in ninhydrin overnight. The membrane was deeper purple than the untreated indicating for this test increased amino functionality. The competitive Pall nylon membrane, treated in the same manner, evidenced a light purple coloration, also indicating an increased amino functionality compared to the untreated membrane.

Detailed Description Text (150):

C. The membrane of Example IA treated with caustic was treated with Hercules resin R4308, dried, cured, washed and redried. Extractables testing (ASTM D 3861-79)



Detailed Description Text (152):

A. Microporous nylon membrane prepared in accordance with Example IA was treated with a Hercule. R 4308 primary charge modifying agent (pH of bath adjusted to 10 with sodium hydroxide) and, where indicated with a polyamine secondary charge modifying agent.

Detailed Description Text (153):

Flow characteristics of the respective membranes showed little or no differentiation, as set forth in Table VII:

Detailed Description Text (154):

B. Filtration performance of the treated <u>membranes</u> was determined in a <u>membrane</u> life test, employing 0.109 monodisperse polystyrene latex from Dow Diagnostic (MDL) mixed with double glass distilled water to produce a dispersion with a turbidity of 10 NTU as measured on a Hach Model 2100A Turbidimeter. The pH of the test dispersion was 7.0.

Detailed Description Text (155):

Flow of contaminated dispersion at a rate of 14 ml/min. was established through a 47 mm. membrane test disc, and differential pressure and turbidity of effluent monitored. The test was deemed complete at 5 psid or attainment of 5 NTU contamination in the effluent. The performance is recorded as elapsed time. Results are set forth in Table VIII, as follows:

Detailed Description Text (156):

C. Filtration performance was also compared between samples of membrane treated with a bath containing either 4.24 wgt. % R 4308 or 2.45% R 4308, both followed by a bath of 0.03% tetraethylene pentamine. The two <u>membranes</u> performed equivalently in <u>membrane</u> life test with 0.109 MDL contaminant challenge. The latter membrane, which contained less primary charge modifier, performed better in dye retention tests.

Detailed Description Text (157):

D. The membranes were tested for extractables, in accordance with ASTM D-3861-79. The results are set forth in Table IX.

Detailed Description Text (161):

FIG. 1 is a graphical representation of Filtration Efficiency (%) versus the Specific Volume Filtered (ml/cm.sup.2) for a charge modified membrane of this invention ("Treated Membrane") and a noncharge modified membrane ("Untreated Membrane"). Both membranes had a 0.2 micrometer rating prior to treatment. The charge modified membrane was produced by treating a nylon microporous membrane with a bath of 2% by weight of Hercules R4308 resin at a pH of 10.5 followed by a bath of 0.1% tetraethylene pentamine. Both the treated and untreated membranes were challenged with a 0.109 MDL contaminant at 0.5 gpm/ft.sup.2.

Detailed Description Text (163):

FIGS. 2 and 3 are Scanning Electron Microscope (SEM) photos of a charge modified membrane produced and challenged as described in this Example VII A with a 0.14 micron MDL beads (FIG. 2-7000 X; FIG. 3-14,000 X).

Detailed Description Text (165):

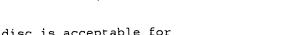
A series of tests were conducted to investigate the characteristics of the charge modified membrane of this invention and process for producing such membrane.

Detailed Description Text (166):

The membrane used for this test series was a single sheet of unmodified, double layer, 0.2 um nylon membrane produced pursuant to the aforementioned Marinaccio et al patent (see Example IA). Each Sample group consisted of three (3) adjacent 21.6 cm.times.27.9 cm sheets, i.e. sheets "A", "B" and "C". The "A: sheet was left untreated and was used to provide data for "unmodified" or "before treatment" membrane. The "B" and "C" groups were subjected to the treatment modes given in Table XI. The "A" and "B" groups were subjected to the following measurements and tests:

Detailed Description Text (175):

Samples 2, 4, 6, 8, 11, 12, 13 and 14, after treatment by the indicated mode exhibited a decrease in flow, i.e. the membranes exhibited clogged pores. This suggests that treatment with the polyamido-polyamine epichlorohydrin resins with a pH as diluted, i.e. less than 7, independent of pretreatment, exhibits pore clogging. After extraction



(which tended to be high--less than 5 mg for 293 mm disc is acceptable for pharmaceutical uses) the flow rates increased indicating a reopening of the pores. Dye adsorption tests indicated the retention of charge after extraction.

Detailed Description Text (176):

Samples 1 and 3, which were treated with trimethylol melamine formaldehyde cationic resin (see aforementioned U.S. Ser. No. 358,822 now abandoned to Ostreicher) and cationic colloidal silica (see aforementioned U.S. Pat. No. 4,305,782 to Ostreicher et al) respectively, showed (a) marginal improvement in dye adsorption tests over untreated membrane, (b) intermediate levels of extraction and (c) even a decrease in dye adsorption after extraction (Sample 3). These tests indicate that the slight charge modification achieved was eliminated after extraction, i.e. the charge modifying agent was not bonded to the surface. Samples 5 and 7 indicate that charge modification, i.e. dye adsorption, after extraction is improved slightly after pretreatment with NaOH solution at pH 10.5, however, the charge modification is vastly inferior to the polyamido-polyamine epichlorohydrin charge modifying agents.

Detailed Description Text (177):

Samples 9 and 10 were both treated with polyamido-polyamine epichlorohydrin resin, Hercules R4308, adjusted to a pH of 10.5. Sample 10 was post-treated with tetraethylene pentamine. Both exhibited no clogging of pores after treatment. Sample 9 exhibited an unexpected enhancement of charge modification, which existed even after extraction over Sample 4 (or even 8) wherein the charge modifying agent was not pH adjusted. Sample 9, does however show an intermediate level of extraction. Sample 10 indicates the same unexpected enhancement of charge modification over Sample 4 (or even 8) but not as high. However, the extraction level is unexpectedly decreased to even below the untreated membrane (Sample 5) due to the post-treatment with the polyamine. Thus, preferred treatment mode for minimizing extraction levels is that of Sample 10. A preferred treatment mode for maximizing charge modification is that of Sample 9.

Detailed Description Text (180):

Two layers of wet microporous membrane, made as in Example IA, were laminated together and dried to 20-25% moisture. It has been found that membrane in such a wet, swollen condition absorbs charge modifying agents more efficiently than bone dry membrane.

Detailed Description Text (181):

The doc: .e layer of membrane was then introduced into a 1.25% by weight solution of Hercules R4308. The pH of the bath was 10.5.

Detailed Description Text (182):

This bath was produced by diluting 38 lbs. (17.17 Kg.) of Hercules R4308 resin from its initial 20% by weight concentration to 5%. Five normal (5N) sodium hydroxide solution was then added to raise the pH to 10.5. The solution was then diluted with D.I. water having greater than 150,000 ohm-cm resistivity in a ratio (volume) 2.5:1. The total volume of bath solution was 60 gallons.

Detailed Description Text (183):

The membrane entered the bath of Hercules R4308 at an angle of 30.degree. from the horizontal to prevent bubble entrapment in the membrane which can prevent the charge modifying agent from diffusing into the membrane. The membrane was treated in this bath at a speed of 2.5 feet/min (76.2 cm/min) for a length of 4 feet (121.9 cm).

Detailed Description Text (184):

Upon exiting this bath, the membrane was wiped on the bottom surface to remove excess water. A 3 minute air soak with cool air movement was used before the membrane entered the secondary charge modifying agent bath.

Detailed Description Text (185):
This bath was produced by adding 0.023% tetraethylene pentamine by weight or 0.113 lbs. (0.0513 kg) to 60 gallons (227. liters) of D.I. water (at least 150,000 ohm-cm resistivity). The pH was about 9. The immersion conditions are identical to the first bath of primary charge modifying agent. The membrane was then wrapped around a take up roll.

Detailed Description Text (186):

The take up roll of wet membrane was stored for at least 3 hours. The roll was then dried at 250.degree. F. (121.degree. C.) for 3 minutes to complete the reaction of the charge modifying agents.

<u>Detailed Description Text</u> (187): The <u>membrane</u> was then washed in a subsequent operation and checked for extraction levels.
Detailed Description Text (189): Membranes treated
Detailed Description Text (192): These <u>membranes</u> are microporous polyvinylidene difluoride, see Grandine II (U.S. Pat. Nos. 4,203,847 and 4,203,848).
Detailed Description Text (202): (3) Millipore Celotate-EH-cellulose acetate, 0.5 micron, were all treated with an aqueous solution containing Hercules R4308 resin, 0.1 to 0.25% (by weight) tetraethylene pentamine and water (qs). The membranes were then dried at 80.degree90.degree. C. for 20-30 minutes. Tests (IBP, FAOP, flow tests and dye adsorption) indicate that significant charge modification had been achieved without deterioration in flow or bubble point characteristics.
Detailed Description Paragraph Table (2): TABLE I Membrane Membrane 4.83 Thitial Bubble Point (psi) 43.7 44.7 44.7 Foam-All-Over-Point 55.0 54.0 54.7 (psi) Thickness Normalized 7.1 7.2 7.0 Flow Rate (cc. mil/min. cm.sup.2 .psi) BET, N.sub.2 adsorption 13.12 13.58
Detailed Description Paragraph Table (3): TABLE II
Detailed Description Paragraph Table (4): TABLE III Endotoxin (pg/ml) Filter Level (pg/ml) 10 ml 50 ml 100 ml Dried, treated 15000 1000 1000 Membrane Control- 15000 10000 10000 10000 Untreated is "picogram")
Detailed Description Paragraph Table (5): TABLE IV Filtrate Efficiency (%) 99.997 Membrane Control-untreated 250000 26.4
Detailed Description Paragraph Table (8): TABLE V: MEMBRANE CHARACTERISTICS THICKNESS NORMALIZED TREATMENT FLOW .sup.3 CHARGE MODIFIER IBP.sup.1 FAOP.sup.2 (cc. mil.sup.2 / PRIMARY SECONDARY SEQUENCE (psi) (psi) min cm.sup.2 psi) None None
48.7 52.0 6.76 2.0% 0.133% Primary 47.3 52.3 6.76 Anquamine.sup.4 First 2.0% 0.133% Secondary 45.3 50.3 6.45 Anquamine First 2.0% 0.133% Mixed 48.7 51.3 6.05 Anquamine.sup.4 2.0% None 46.7 51.0 7.25 2.0% 0.03% Primary 49.5 54.0 5.80 Tetraethylene First Pentamine
Initial Bubble Point .sup.2 Foam All Over Point ##STR10## an empirically derived relation to normalize data for thickness variationssup.4 Anguamine 100, a low molecular weight (under 10,000) cationic polyamide adduct evidencing secondary amine functionality by comparative UV spectroanalysis, supplied by Pacific Anchor Chemical Corp.
Detailed Description Paragraph Table (12): TABLE XI TREATMENT MODES NO. PRETREATMENT CHARGE MODIFICATIONS POST TREATMENT 1 NONE Pares
607 Colloid-2 wt. % Drain and stretch solution PH as diluted dry 2 NONE 1884 Resin - 2 wt. % solu-Drain and stretch tion -pH as diluted dry 3 NONE Wesol PA - 2 wt. % solu-Drain and stretch tion - pH as diluted dry 4 NONE 4308 Resin - 2 wt. % solu-Drain and stretch tion - pH as diluted dry 5 Soak membrane in Parez 607 Colloid - 2wt. Drain and stretch dilute NaOH solution % solution - pH as diluted dry (pH 10.5) for two 6

minutes. Drain and 1884 Resin - 2 wt. % solu- Drain and stretch oven dry. Wash in tion -pH as diluted dry distilled water 7 Wesol PA - 2 wt. % solu- Drain and stretch tion - pH as diluted dry 8 4308 Resin - 2 wt. % solu- Drain and stretch tion - pH diluted dry 9 NONE 4308 Resin - 2 wt. % solu- Drain and stretch tion - pH diluted dry 10 NONE 4308 Resin - 2 wt. % solu- 0.03 wt % solution Pentamine tion pH adjusted to 10.5 Drain and stretch dry 11 0.03 wt. % solution 4308 Resin - 2 wt. % solu- Drain and stretch dry Pentamine tion pH as diluted 12 0.03 wt. % solution 1884 Resin - 2 wt. % solu- Drain and stretch dry (1.4 butanediol) tion - pH as diluted 13 0.03 wt. % solution 4308 Resin - 2 wt. % solu- Drain and stretch dry (1.4 butanediol) tion as diluted 14 0.03 wt. % solution 1884 Resin - 2 wt. % solu- Drain and stretch dry DGE (1.4 butanediol) tion -pH as diluted 15 NONE NONE - soak in water Drain and stretch dry Parez 607

melamine formaldehyde cationic colloid 1884 Resin Hercules 1884 polyamidopolyamine epichlorohydrin Wesol PA cationic colloidal silica 4308 Hercules Inc. R 4308 polyamidopolyamine epichlorohydrin resin.

Detailed Description Paragraph Table (15):

Dye Adsorption - Extraction IBP FAOP Q(ml/ Time To (mg per 47 Sample (PSI) (PSI) min) Breakthru(Min) mm disc) GVHP 3.0 5.0 0(at 15 Not Run 0.01 untreated

PSID) GVHP 4.0 15.0 0(at 15 Not Run 0.14 treated PSID) GVWP 51.0 53.5 72.0 1 0.47 untreated GVWP 50.0 54.0 75.0 56.0 0.18 treated

Q -- flow of water Conclusion: Hydrophobic polymer membranes are not amenable to charge modification by the methods of this inventions whereas hydrophilic versions of the same polymer are amenable to charge modification.

Other Reference Publication (1):

Knight, R. A. et al., "Microporous Membranes as Depth Filters", World Filtration Congress III, 9/13 to 17/1982.

Other Reference Publication (7):

Walterwinek, Electronic and Chemical Aspects of Water Filtration, Filtration and Separation, May/Jun. 1974.

CLAIMS:

- 1. A process for the filtration of a liquid having particulate contaminants therein comprising passing the liquid through a symmetrical, isotropic porous hydrophilic cationic charge modified microporous filter membrane comprising:
- a symmetrical, isotropic porous hydrophilic organic polymeric microporous filter membrane having a microstructure throughout said membrane and a pore size of at least 0.05 microns; and
- a charge modifying amount of a primary charge modifying agent bonded to substantially all of said membrane microstructure, without substantial pore size reduction or pore blockage, wherein the primary charge modifying agent is a water soluble organic polymer having a molecular weight greater than about 1000, wherein each monomer thereof has at least one epoxide group capable of bonding to the microstructure of the membrane and each monomer thereof has at least one tertiary amine or quaternary ammonium group,

whereby the contaminants are removed from the liquid by retention by said cationic charge modified membrane.

- 3. The process of claim 1 or 2, wherein the hydrophilic organic polymeric microporous membrane is polyvinylidene fluoride.
- 4. The process of claim 1 or 2, wherein the hydrophilic organic polymeric microporous membrane is an ester of cellulose.
- 5. The process of claim 1 or 2, wherein the hydrophilic organic polymeric microporous membrane is nylon.
- 6. The process of claim 1 or 2, wherein the hydrophilic organic polymeric microporous membrane is polyhexamethylene adipamide.
- 7. The process of claim 1 or 2, further comprising sanitizing or sterilizing the charge modified membrane prior to passing the liquid through the membrane.

12. The process of claim 11, wherein the hydrophilic organic polymeric microporous membrane is polyhexamethylene adipamide.

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Search Results - Record(s) 1 through 2 of 2 returned.

1. Document ID: US 6029479 A

L5: Entry 1 of 2

File: USPT

Feb 29, 2000

US-PAT-NO: 6029479

DOCUMENT-IDENTIFIER: US 6029479 A

TITLE: Fine particle lint filter

DATE-ISSUED: February 29, 2000

INVENTOR-INFORMATION:

NAME

CITY

STATE

ZIP CODE

COUNTRY

Pattee; Harley J.

Orlando

FL

32821

US-CL-CURRENT: 68/18F; 210/314, 210/335

Full Title Citation Front Review Classification Date Reference Sequences Attachments Claims KWC

2. Document ID: US 5436384 A

L5: Entry 2 of 2

File: USPT

Jul 25, 1995

US-PAT-NO: 5436384

DOCUMENT-IDENTIFIER: US 5436384 A

TITLE: Process for the remediation of contaminated particulate material

DATE-ISSUED: July 25, 1995

INVENTOR-INFORMATION:

NAME

CITY

STATE

ZIP CODE

COUNTRY

Grant; David C.

Gibsonia

PA

Lahoda; Edward J.

Edgewood Borough

PA

Keegan; C. P.

South Huntington Twp.

US-CL-CURRENT: 588/1; 209/17

Citation Front Review Classification Date Reference Sequences Attachments Claims KWC Draw, Desc Image

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Terms	Documents
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<u>Previous Page</u> <u>Next Page</u>

L10: Entry 4 of 6

File: USPT

May 31, 1994

DOCUMENT-IDENTIFIER: US 5316751 A

TITLE: Methods for mine tailing clean-up using recovery technologies

Brief Summary Text (3):

In the mining industry milling of mined rock is frequently done proximate to water supplies, since typically large quantities of water are used in the milling process to develop a suitable concentrate for refining operations. Mills are often centrally located to receive ores from a variety of different mine sources such that the composition of discarded material is often complex, having a variety of different potential contaminants. During the milling operation, ore is customarily crushed to a fine consistency, such that discarded tailings include primarily fine sands and slimes. Slimes are generally characterized as material of a particle size that remains suspended in water for a period of time and are usually less than 200 mesh in screen size.

Brief Summary Text (17):

Cementation will be directed at the formulation of different binders primarily from on-site processed materials for the production of conventional lime based cements, fibre impregnated cements, sulfur based cements and geopolymer cements, and, the use of such binders in the fabrication of on-site refining, reclamation and containment structures and saleable off-site building products, including cultured stone, tiles and precast structural materials. Reprocessed tailings can be used as composition fill material or classified and refined for use in glass or other vitrified products.

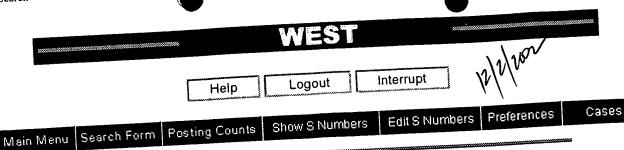
Detailed Description Text (3): Referring to the process flow diagram, the tails 10 are transported to a portable wash trough 12 by front-end loaders 14 and conveyed as a wet cake to a wash screen 16 for elimination of oversize material. A cleaned and screened slurry is drawn from the screen basin 18 and pumped to a classifying cyclone 20 with the over 200 mesh material from the underflow pumped to a high volume cyclone 22 for specific gravity separation of the plus 200 mesh material at a 3.5 cut to remove sulfides of iron, lead and zinc and other heavy metals before the plus 200 mesh material is fed to a slugger 24 to partially dewater before deposit of these sands to dry storage 26. The heavies from the plus 200 mesh material is ground in a bar mill 28 and returned to the wash basin for reclassification. Overflow from the screen basin and discharge water from the slugger are piped to a thickener or decanting pond 29. Settled material, which is well under 200 mesh, is periodically returned to the circuit line for minus 200 mesh material.

Detailed Description Text (15):

The discharge from this stream together with the discharge from the FCL subcircuit will contain the major toxins not previously collected in the calciner condensers. These materials are the primary candidates for geopolymer cementation or glass vitrification to bind the toxins into inert block. Alternately, the material with cadmium and arsenates removed is discharged to a receiving pond 45, can undergo a staged biological detoxification in the waste water treatment stream.

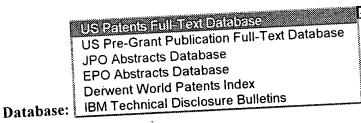
Detailed Description Text (30):

The chamber segments 104 are separated by a screen or fibre filter 118 and a membrane filter 120 of pore size to inhibit migration of bacteria when desired, for example between anaerobic and aerobic chamber sections. As shown in FIG. 5, water jets 122 can be employed to add water, with or without nutrients to the lower confines at the chamber and move sediments to the collection tube in stages where at least one set of filters are removed. Gas jets 124 can also be employed to add gases, such as CO.sub.2 to the water to promote bacteria growth.



Search Results -

Search	
Terms	Documents
L9 and filter	17



Search:

L10		E	Refine Search
	Recall Text 🗢	Clear	

Search History

DATE: Tuesday, December 03, 2002 Printable Copy Create Case

et <u>Name</u>	Query	Hit Count Se	et Name esult set
de by side			
DB=US	SPT; PLUR=YES; OP=ADJ	17	<u>L10</u>
<u>L10</u>	L9 and filter	33	<u>L9</u>
<u>L9</u>	L8 and screen	89	<u>L8</u>
<u>L8</u>	rotary drum and sprayer	. 0	<u>L7</u>
<u>L7</u>	dual sprayer and rotary drum and screer	1	<u>L6</u>
<u>L6</u>	15 and spraying	15	<u>L5</u>
<u>L5</u>	L4 and reverse osmosis	222	<u></u> <u>L4</u>
<u>L4</u>	L3 and membrane	671	<u>L3</u>
<u>L3</u>	filter and screen and meshes and hair	0	<u></u> <u>L2</u>
<u>L2</u>	L1 and screenand filter and hair	672	L <u>l</u>
<u>L1</u>	((210/767)!.CCLS.)	072	<u> </u>

END OF SEARCH HISTORY

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	Generate Collection Print	12/2/2001	
: Entry 1 of 15	File: USPT		Nov 5, 2002

DOCUMENT-IDENTIFIER: US 6474111 B1

TITLE: Recycling system for laundry wash water

Abstract Text (1):

A recycling system for laundry wash water having a collection tank for collecting used wash water. A holding tank connected to the collection tank has heat exchange piping in the interior of the holding tank. A lint/grit separator is connected to the holding tank. A lint filter acting as a second stage in lint removal is connected to the holding tank. An oil separator for removing free oil is connected to the lint filter. A media filter having a medium of fine rock is connected to the oil separator. A hydrocarbon absorption filter is connected to the media filter. An activated carbon filter is connected to the hydrocarbon absorption filter. A final holding tank is connected to an ozone generator so that clean and processed water leaves the final holding tank. Clean and processed water leaving the final holding tank is channelled to the heat exchange piping in the holding tank which uses the used wash water from the collection tank to heat the clean and processed water before the clean and processed water goes on to a hot water server connected to a wash machine facility.

Brief Summary Text (16):

A lint/grit separator for removing suspended solids and lint from the used wash water is connected to the holding tank by means of piping. Lint and grit exceeding a specific size are removed from the used wash water by the lint/grit separator so that lint-grit separated water proceeds forward to a lint <u>filter</u>. Suspended solids and lint which exceed a predetermined size fall from the lint/grit separator to a lint/grit collection basket.

Brief Summary Text (17):

The lint <u>filter</u> receives the lint/grit separated water and removes smaller-size lint and grit from the lint-grit separated water. Having passed through the lint filter, lint-filtered water exits the lint filter and continues to an oil absorption filter.

Brief Summary Text (18):

The oil absorption filter removes free oil having a density less than water from the lint-filtered water. Upon passing through the oil absorption $\underline{\text{filter}}$, the lint-filtered water becomes oil-filtered water and exits the oil absorption $\underline{\text{filter}}$ through piping to a media filter.

Brief Summary Text (19):

Upon the oil-filtered water entering the media filter, suspended solids and various hydrocarbons are removed from the oil-filtered water so that media-filtered water exits the media filter and is channelled to a hydrocarbon absorption filter.

Brief Summary Text (20):

The hydrocarbon absorption $\underline{\text{filter}}$ removes emulsified hydrocarbons and some heavy metals from the media-filtered water so that absorption-filtered water exits the hydrocarbon absorption filter through piping to an activated carbon filter.

Brief Summary Text (21):

The activated carbon filter removes remaining organic compounds including surfactants and soap from the absorption-filtered water so that clean and processed water exits the activated carbon filter.

Brief Summary Text (22):

The clean and processed water upon exiting the activated carbon filter enters a final holding tank where it is connected to piping which branches in two--with one branch

exiting to a cold water server located outside the recycling system and the other branch connecting to the heat exchange piping in the interior of the holding tank.

Brief Summary Text (24): The collection tank, holding tank, separator, lint $\underline{\text{filter}}$, oil $\underline{\text{filter}}$, media $\underline{\text{filter}}$, absorption $\underline{\text{filter}}$, carbon $\underline{\text{filter}}$ and final holding tank are to be viewed as elements of the recycling system of the present invention.

 $\frac{\text{Drawing Description Text}}{\text{FIG. 9 is a perspective, partial see-through view of the lint } \underbrace{\text{filter}}_{\text{filter}} \text{ of the present invention:}$

 $\frac{\text{Drawing Description Text}}{\text{FIG. 10 is an exploded see-through view of the lint } \underbrace{\text{filter}}_{\text{of the present invention and depicts the grooved guides for placing the removal }}_{\text{screens}};$

 $\frac{\text{Drawing Description Text}}{\text{FIG. 11 is a schematic illustration depicting the angular relation of the lint removal}}{\text{screens}} \text{ to the spray nozzle and to the bottom of the lint } \frac{\text{filter}}{\text{filter}};}$

 $\frac{\text{Drawing Description Text}}{\text{FIG. 12 is a perspective illustration of the spray bar and nozzle of the lint <math>\frac{\text{filter}}{\text{of the present invention}}$;

Drawing Description Text (19): FIG. 13 is a see-through perspective view of a preferred oil absorption filter according to the present invention;

Drawing Description Text (20): FIG. 14 is a see through perspective view of the oil absorption <u>filter</u> of FIG. 13 with the hydrophobic socks removed;

 $\frac{\text{Drawing Description Text}}{\text{FIG. 15 is a see-through side view of the oil absorption }} \underbrace{\text{filter of FIG. 13}}_{\text{13}};$

 $\frac{\text{Drawing Description Text}}{\text{FIG. 16 is a see-through perspective view of the media}} \;\; \frac{\text{filter}}{\text{filter}} \;\; \text{of the present invention;}$

Drawing Description Text (23): FIG. 17 is a see-through view of the bottom of the media <u>filter</u> viewed from the top;

Drawing Description Text (24): FIG. 18 is a see through view side-view of the media <u>filter</u> filled with igneous rock;

Drawing Description Text (25): FIG. 19 is a schematic illustration of the overflow column in the media <u>filter</u> of the present invention;

<u>Drawing Description Text</u> (26): FIG. 20 is a perspective, partial see through view of the hydrocarbon absorption <u>filter</u> according to the present invention;

Drawing Description Text (31):
FIG. 24 is a schematic illustration of a reverse osmosis system for use with the recycling system of the present invention in conjunction with a dish washing facility;

Detailed Description Text (6):
Ozone traveling from ozone generator 60 is injected into the stream of lint/grit separated water W.sub.lg traveling through pipeline 49 as it connects to pipe 61 connected to the ozone generator 60. The lint/grit separated water then enters pipeline 62 before being introduced to lint filter 64.

 whereupon the lint/grit separated water is further processed so as to become lint-filtered water W.sub.lf before entering pipeline 79.

Detailed Description Text (8):

Through pipeline 79 lint-filtered water W.sub.lf is channelled to oil absorption filter 80 which removes oils having a density less than water such that the lint filtered water W.sub.lf becomes oil filtered water W.sub.of.

Detailed Description Text (9):

Pipeline 104 serves as the link between oil absorption <u>filter</u> 80 and media <u>filter</u> 105 and channels the oil filtered water from absorption filter 80 to media filter 105.

Detailed Description Text (10):

The Media $\underline{\text{filter}}$ 105 removes suspended solids and various hydrocarbons such that the oil filtered water W.sub.of becomes media filtered water W.sub.mf. The media filtered water W.sub.mf exits the media filter 105 through pipeline 123 which connects to centrifugal pump 124. After entering centrifugal pump 124 the media-filtered water is injected with ozone from ozone generator 127, which connects to the piping 125 leading from centrifugal pump 124, through pipe 126.

Detailed Description Text (11):

The media filtered water W.sub.mf having received an injection of ozone from ozone generator 127 proceeds to the hydrocarbon absorption filter 130 through pipeline 128.

Detailed Description Text (12):

Hydrocarbon absorption filter 130 removes emulsified hydrocarbons and some heavy metals from the media filtered water received from media filter 105 such that the media filtered water W.sub.mf becomes absorption filtered water W.sub.af.

Detailed Description Text (13):

From the hydrocarbon absorption filter, the absorption filtered water W.sub.af is channeled through pipeline 134 to activated carbon filter 140.

Detailed Description Text (14):

Activated carbon filter 140 is designed to remove any remaining organic compounds such that the absorption filtered water becomes carbon filtered water W.sub.cf.

Detailed Description Text (15):
The carbon filtered water W.sub.cf exits the activated carbon <u>filter</u> 140 through pipeline 150 which connects to final holding tank 156.

Detailed Description Text (16):

The final holding tank 156 is connected by pipe 157 to an ozone generator 158 through which more ozone is injected into the carbon filtered water received from the activated carbon_filter 140. Tap water 8 is introduced into final holding tank 156 through pipeline 9 to recoup water lost in the recycling process. Clean and processed water W.sub.cp exits final holding tank 156 through pipeline 162.

Detailed Description Text (53):

The lint/grit separated water W.sub.lg, having passed through the lint/grit separator 42 and into pipeline 49, is injected with ozone by ozone generator 60 (FIGS. 1 and 2) before proceeding through pipeline 62 to lint filter 64. The ozone causes the organic contaminants in the lint/grit separated water to separate and serves as a disinfectant.

Detailed Description Text (54):

The ratio of the lint/grit separated water W.sub.lg which continues from the lint/grit separator 42 toward lint filter 64 as compared to the water W.sub.rw returned to the collection tank 12 is approximately 75 to one.

Detailed Description Text (55):

With reference to FIGS. 9, 10, 11 and 12, lint/grit separated water W.sub.lg enters lint filter 64 through entrance 63. Upon entering the lint filter 64 the lint/grit separated water W.sub.lg begins a trek through the lint filter which serves as a second stage of lint removal.

Detailed Description Text (56):

Lint filter 64 is provided with a series of mesh screens 66A, 66B and 66C. Each successive screen is provided with mesh of decreasing size. The screens are fitted into grooved slats 70 provided on one side 69 of the lint $\underline{\text{filter}}$ (FIG. 10) and into corresponding grooved slats provided on an opposite side of the lint $\underline{\text{filter}}$ to side 69.

Detailed Description Text (57):

Alternatively, the sides of the lint filter can be made to have grooves built into its opposite sides for accommodating the mesh screens 66; or, guide tabs 71 can be placed on the posite side of the lint filter as portrayed in FIG. 11; or other manners to secure the mesh screens to the sides of the lint filter may be used. The mesh screens extend from just below the top of the lint filter to the bottom of the lint filter.

Detailed Description Text (58):

Upon entering entrance 63 the lint/grit separated water W.sub.lg enters a spray bar 65 which branches out laterally so that a plurality of spray nozzles 73(FIG. 12) evenly distribute the lint separated water W.sub.lg downward to the water level W.sub.l in the lint filter 64.

Detailed Description Text (59):

The screens 66 are arranged in parallel relation to each other but are angled forward toward entrance 63 to form an angle .THETA. of 45 to 90 degrees in relation to the bottom 69 of the lint filter 64 as depicted in FIG. 11.

Detailed Description Text (60):

The lint contained in the lint/grit separated water is not allowed to leave the lint filter because small diameter lint is trapped by the screens 66.

Detailed Description Text (61):

The screens being arranged at an angle, most of the lint settles to the bottom, making cleaning of or replacement of the screens an easier task. Cleaning of the screens is done by taking off a removable top (not shown) of the lint filter 64.

Detailed Description Text (62):

Exits 72A, 72B and 72C are positioned below water level W.sub.l in the lint filter 64. Lint-filtered water W.sub.lf travels through the exits 72A, 72B and 72C and to corresponding pipelines 79A, 79B, 79C to the oil filter 80 (FIGS. 14 and 15).

Detailed Description Text (63):

The multiple pipes 72A, 72B, 72C result in a slow flow of water so that oil is inclined to separate from the lint filtered water as it enters the oil filter 80.

Detailed Description Text (64):

With reference to FIGS. 13, 14, and 15, lint-filtered water W. sub.lf, enters oil filter tank 80 through entrance connections 82A, 82B and 82C which are connected to pipelines 79A, 79B and 79C which connect the lint filter 64 to the oil filter. The oil filter 80 is provided with an upper platform 85, a middle platform 87 and a bottom platform 89 which are used to secure rows of cylindrical hydrophobic socks 84A.sub.1, 84A.sub.2 . . . 84B.sub.1, 84B.sub.2 . . . , etc.

Detailed Description Text (65):

The socks are made of 100% reclaimed wood fiber or other suitable hydrophobic material. The oil absorbing socks 84, being hydrophobic, allow water to pass by while absorbing free ci... The socks can be replaced by removing a removable top (not shown) of the oil filter.

Detailed Description Text (67):

Each hole 88 is provided with four slot guides 86 which are thin and stick shaped and which extend through each platform to easily guide the socks through the holes 88. The bottom platform 89 sits atop the bottom 90 of the oil filter 80. Thus, each hydrophobic sock extends through each platform 85, 87 and 89 and touches the bottom 90 of the oil filter 80.

Detailed Description Text (68):

The oil filter is formed and defined by sides 91, 92, 93, 94 and bottom 90. Side 91 is opposite to side 92 and side 93 is opposite to side 94.

Detailed Description Text (69):

The platforms 85, 87, 89 of oil filter 80 extend from side 91 of the oil filter 80 but do not extend all the way across to the other side 92 of the oil filter 80 allowing water flow between the platforms 85, 87 and 89.

Detailed Description Text (70):

A pumping chamber 100 is partitioned off from the rest of the interior components of the oil <u>filter</u> 80. Pumping chamber 100 lies in the corner of oil <u>filter</u> 80 and is formed by partition wall 101 and partition wall 102 which connect to each other and to walls 93 and 92 of oil filter 80, respectively. Platforms 85, 87, 89 connect to partition walls 101 and 102 and connect to sides 91, 92 and 94, but do not connect to side 93 of the oil filter 80 so as to create a flow channel between platform levels.

Detailed Description Text (71):

The lint filtered water W. sub.lf which enters the oil absorption <u>filter</u> 80 proceeds through entrances 82A, 82B and 82C proximate to the level of the middle platform 87 and below the water level in the oil absorption filter.

Detailed Description Text (72):

Upon entering the interior of the oil filter 80, free oil having a density less than water rises to the top of the lint filtered water W.sub.lf.

Detailed Description Text (74):

Oil-filtered water W.sub.of after having traveled around the rows of oil absorption socks 84 has but one path of travel to reach the pumping chamber 100. That path is provided through pipe or opening 97 which is located near the bottom of the oil filter 80 and which extends through partition wall 102.

Detailed Description Text (75):

As the oil-filtered water W.sub.of enters the pumping station 100 through pipe 97 the water level in the pumping station rises. As the water level rises, a float switch 98 is activated as float 96 rises upward on a guide rod. As float switch 98 activates, sump pump 95 is energized causing oil filtered water W.sub.of to be pumped into pipe 103. Oil filtered water W.sub.of is pumped from the sump pump 95 into pipe 103 and out of the oil filter through pipeline 104 to media filter 105.

Detailed Description Text (76):

With reference to FIGS. 16 and 17, oil-filtered water W.sub.of traveling through the pipeline 104 enters distribution piping 106 which extends around the top periphery of media <u>filter</u> 105 just inside walls 111A, 111B, 111C and 111D of the media <u>filter</u> 105.

Detailed Description Text (77):

The size of the media <u>filter</u> is approximately 4 ft. by 4 ft. by 4 ft. or sized as conditions warrant. In high volume situations, an additional media filter could be utilized.

Detailed Description Text (78):

The media filter 105 is constructed to hold a medium 110 for removing contaminants such as suspended solids and hydrocarbons from the oil filtered water W.sub.of received from oil filter 80. The medium 110 contained in the media filter is igneous rock and/or sand from 1/8 inch in diameter to two inches in diameter or a material proven to have at least the same filtering capabilities.

Detailed Description Text (79):

The size of the media rock or sand will depend upon the type of water being recycled. Laundromats and uniform shops tend to have greasier more oily water than do motels, for example. Accordingly, the type of medium which fills the media filter will be a diameter best suited for a given operation. For greasier water, a larger diameter of media rock or sand is desired.

Detailed Description Text (80):

Media Filter Spray nozzles 107 located on the distribution piping 106 spray the oil-filtered water W.sub.of received from the oil filter 80 upon the top of medium 110. The medium 110 extends some three to four feet to the bottom 112 of the media filter 105. At the bottom of the media $\frac{\text{filter}}{\text{filter}}$ lies a collection pipe 113 which is a 3 inch diameter pvc pipe, or other appropriate piping, which is provided with multiple perforations 114.

Detailed Description Text (81):

As the oil filtered water is sprayed on the top of medium 110 by spray nozzles 107, the oil filtered water percolates through the medium 110 until it reaches the bottom 112 of the media filter.

Detailed Description Text (82):

Collection pipe 113 extends diagonally across the bottom 12 of the media filter with an end region 115 of the collection pipe 113 entering a six by six inch square partition column 116 which extends vertically in a corner of the media <u>filter</u>.

Detail Description Text (83):

Upon reaching the bottom of the media $\underline{\text{filter}}$, the oil filtered water W.sub.of is now media filtered water W.sub.mf which collects in pipe 113 and flows into the partitioned column 116 through end region 115 of the collection pipe 113. At the bottom of the partitioned column 116 is located an outlet pipe 117 which connects to pipeline 123 which leads the media filtered water toward the oil absorption filter 130 (FIG. 1).

Detailed Description Text (84):

The partitioned column 116 is provided with an activation switch which is one fourth of the way up the column from the bottom of the media filter. This activation switch turns on centrifugal pump 124 and ozone generator 127 which causes media filtered water W.sub.mf to exit the media $\underline{\text{filter}}$ as a result of the draft created by pump 124.

Detailed Description Text (85):

The top of the partitioned column is located some six or eight inches below the top of the media <u>filter</u> tank and acts as an overflow mechanism in that excess water will flow into the top of the column and will not spill out of the media tank.

Detailed Description Text (86):

With reference to FIG. 20 and FIG. 1, the media filtered water passes from the media filter 105 to pump 124 to pipeline 125 where it is injected with ozone and proceeds on to hydrocarbon absorption filter 130.

Detailed Description Text (87):
The absorption filter 130 is a pressurized tank approximately six feet tall and 42 inches in diameter and is 2/3 full of organic clay 132.

Detailed Description Text (88):

The media filtered water is pumped at approximately 60 psi up into the top inner region of the tank (not shown) whereupon the media filtered water is further purified of emulsified hydrocarbons and some heavy metals as it seeps downward through the organic clay 132. Upon reaching the bottom of the absorption <u>filter</u> 130, the media filtered water is now considered to be absorption filtered water W.sub.af which proceeds through pipe 134 toward the activated carbon filter 140.

Detailed Description Text (89):

With reference to FIG. 21, the activated carbon filter 140 is a pressurized tank similar in dimensions to hydrocarbon <u>filter</u> 130. However, activated carbon <u>filter</u> 140 is 2/3 filled with virgin activated carbon.

Detailed Description Text (90):

When the recycling system is in operation, the absorption filtered water W.sub.af travels through pipe 134 which becomes pipe 135. The absorption filtered water W.sub.af proceeds through pipe 135 and through open solenoid switch D which being open allows the water W.sub.af to pass into pipe 139 which distributes the absorption filtered water to the top and inside of the carbon filter tank 140. The hydrocarbon filtered water passes downward through the virgin $1\overline{42}$. carbon to the bottom where it enters pipe 143.

Detailed Description Text (93):

When the activated carbon filter 150 is backwashed after a daily operation, for instance, during the evening when the system is not in use, solenoid switches B and D are closed and the solenoids A and C are open for purposes of backwashing filter 150.

Detailed Description Text (95):
The carbon filtered water W.sub.cf leaves the carbon activated <u>filter</u> through pipeline 150 to final holding tank 156.

Detailed Description Text (109):

The oil separator 80 is provided with a level or float switch 98 in its containment compartment 100 which sends a signal to the control panel 300 to turn on sump pump 95 when the containment compartment 100 in the oil $\underline{\text{filter}}$ 80 is 2/3 full. Upon the sump pump turning on and the water level in the containment compartment going down as a result, the float switch 98 sends a signal to the control panel 300 to turn off sump

pump 95 when the level in the containment compartment is 1/2 full.

Detailed Description Text (110):

In the containment chamber 116 of the media $\underline{\text{filter}}$ 105 a float switch 120 is provided which sends a signal to control panel 300 to turn on centrifugal pump 124 and ozone generator 127 when the water level in containment chamber 116 is more than 1/4 full.

Detailed Description Text (111):

Water from the containment chamber of the media <u>filter</u> is then drawn toward the hydrocarbon filter tank 130 as a result of the activation of pump 124. When the recycling system is in operation the water from the hydrocarbon <u>filter</u> tank enters carbon filter tank 140.

Detailed Description Text (112):

This is made possible by the control panel 300 being connected to solenoid switches B and D open so that absorption filtered water W.sub.af can travel through pipe line 134 into carbon filter tank 140 and allowing carbon filtered water W.sub.cf to pass from the carbon filter 140 onto the final holding tank 156.

Detailed Description Text (113):

When the system is not in use, solenoid switches B and D are closed by timers on the control panel, and solenoid switches A and C are open so that the carbon filter tank can be backwashed.

Detailed Description Text (114):

The carbon filtered water W.sub.cf which leaves the carbon filter 140 is injected with ozone in the final holding tank 156. Ozone generator 158 is always on when the recycle system is in operation and has its own built-in compressor which connects to control panel 300.

Detailed Description Text (116):

With reference to FIG. 24 and FIG. 1, the reverse osmosis system 200 for use with the recycling system of the present invention is provided with a cartridge filter 202 which connects pump 204 through pipe 203.

Detailed Description Text (117):

From pump 204, the clean and processed water W.sub.cp enters pipe 205 and onto pipe 208 which becauses such that hyperfiltration filters 206 and 212 are arranged in parallel.

Detailed Description Text (118):

Hyperfiltration filters are taught in U.S. Pat. No. 5,639,374. Only mineral-free clean and processed water W.sub.cp enters the composite membrane pipe (not shown) located in the center region of the hyperfiltration filters 206, 212.

Detailed Description Text (119):

From the composite membrane pipe of filters 206, 212, clean soft water W.sub.cs suitable for washing dishes leaves filters 206 and 212 through pipe 214 which connects to a dishwater heater (not shown).

Detailed Description Text (120):

The clean and processed water not allowed into the composite membranes of filters 206, 212 exits the filters through pipe 210 for re-introduction into the recycling system 10.

Detailed Description Text (123):

Thus, the lint/grit separator 50 can be viewed as a step in the recycling system in which lint and grit which exceed a certain size are removed from the used wash water. The lint/grit separated water W.sub.lg having gone through the lint/grit separator continues to the lint filter 64.

Detailed Description Text (124):

However, the lint/grit separated water is injected with ozone by ozone generator 60 before proceeding to the step of entering lint filter 64.

Detailed Description Text (125): Lint <u>filter</u> 64 removes lint, threads, <u>hair</u> and other fine elements from the lint/grit separated water. Having passed through the step of ozone injection and through a second lint removal step by lint filter 64, the lint filtered water W.sub.lf continues to oil

separator 80.

Detailed Description Text (126):
In oil . parator 80, free oil is removed from the lint filtered water W.sub.lf. Thus, oil separator 80 performs an oil removal step so that oil separated water W.sub.os proceeds to a media filtration step in media filter 105. Media filter 105 removes suspended solids and remaining hydrocarbons in the oil separated water W.sub.os as a result of the oil separated water proceeding through the igneous rock and/or sand of the media filter.

Detailed Description Text (127):

Upon passing through the media filter 105, the media filtered water W.sub.mf is further treated to an ozone injection before entering the hydrocarbon absorption filter 130 where the media filtered water as a result of the water pressure created by pump 124 is forced through the clay in the hydrocarbon absorption filtered.

Detailed Description Text (128):

This step of clay filtration of the media filtered water results in hydrocarbon filtered water W.sub.hc passing from the hydrocarbon absorption filter into the activated carbon filter 140 where soap in the hydrocarbon filtered water W.sub.hc is effectively removed.

Detailed Description Text (129):

The carbon filter 150 subjects the hydrocarbon filtered water to a carbon filtration step so that carbon filtrated water W.sub.cf passes into the final holding tank 156 where the carbon filtered water is further subjected to a final ozone injection step before leaving the final holding tank as clean and processed water W.sub.cp.

Detailed Description Text (131):

If the recycling system of the present invention is to be used to recycle water from dish washer(s), a reverse osmosis step is called for which takes place in the reverse osmosis system 200 before introduction to a dishwater heater.

Detailed Description Text (132):

The collection tank 12, holding tank 20 and heat exchange piping 26, lint/grit separator 42, lint filter 64, and oil filter 80, media filter 105, hydrocarbon filter 130, carbon filter 140 and final holding tank 156 are viewed as elements of the recycling system of the present invention.

CLAIMS:

- 1. A recycling system for laundry wash water, comprising: a collection tank for collecting used wash water; a first lint removal means comprising a lint/grit separator for removing lint and grit which exceeds a specific size from the used wash water, said lint/grit separator connecting to said collection tank; a second lint removal means comprising a lint filter connected to said lint/grit separator for removing lint which was not removed by said lint/grit separator; an oil filter for removing free oil from the uses wash water, said oil filter being connected to said lint filter; a media filter for further filtering the used wash water, said media filter being connected to said oil filter; a hydrocarbon absorption filter for further filtering the used wash water, said hydrocarbon absorption filtered being connected to said media filter; an activated carbon filter connected to said hydrocarbon absorption filter; a heat exchanger for supplying heat to the used wash water which has passed through said oil filter; an ozone generator interposed in said system after said lint/grit separator, such that ozone is injected into the used wash water; means for channeling the used wash water which has passed through said lint/grit separator and through said lint filter to a water server, said means for channeling the used wash water connecting to said lint filter, wherein said means for channeling the used wash water to a water server comprises a centrifugal pump; and a final holding tank directly connected to a second ozone generator, said final holding tank being connected to said activated carbon filter and to said centrifugal pump.
- 3. A recycling system according to claim 2, further comprising: a reverse osmosis system connected to said water server.

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L5: Entry 10 of 15

File: USPT

Nov 26, 1996

DOCUMENT-IDENTIFIER: US 5578213 A TITLE: Fluid treatment process

Brief Summary Text (4):

The treatment of wastewater to reduce effluents and recycle usable water is a matter of the utmost importance to the overall effort of reducing pollution and conserving water. A particular area of concern is the handling of graywater aboard ships and boats, e.g., marine graywater. Graywater is defined as the combined effluent wastewaters from shipboard galleys, sculleries, dishwashers, laundries, showers, sinks, and wash basins (including deep sinks in work areas such as machine shops and medical areas). The contaminants are typically food particles of up to substantial dimensions (e.g., food slices), animal fats, vegetable oils, soaps, detergents, body oils, human hairs, metallic particles from sculleries and machine shops, solvents, and small articles of clothing (e.g., socks) which may have snaked their way through the laundry system. Graywater is usually differentiated both from blackwater, which is a salt-water-based collection of body wastes and paper materials from shipboard head spaces, and from bilgewater, which is a salt-water-based accumulation of all other shipboard wastewater and which may contain chemical solvents and the like.

Detailed Description Text (12):

To further facilitate the efficient treatment of the wastewater, the wastewater is preferably passed through a strainer prior to passage through the first filtration medium (and also preferably prior to the removal of free oil, although this is not as significant). Any suitable strainer can be so utilized, e.g., a duplex strainer, and the strainer is designed to remove larger particulate matter (such as hair, laundry items, and the like) and, as such, will typically be a perforated plate or, more preferably, a screen mesh. A suitable screen mesh strainer can be of any suitable configuration, but will preferably have about 10-50 wires per linear inch in each direction, with any suitable wire diameter, e.g., about 0,002-0.02 inch. More preferably, such a screen mesh strainer will have about 20-40 wires per linear inch, most preferably about 30 wires per linear inch, in each direction, with any suitable wire diameter, e.g., about 0.002-0.02 inch, preferably about 0.005-0.01 inch. The strainer desirably removes particles greater than at least about 1000 .mu.m, preferably greater than at least about 600 .mu.m, in diameter.

Detailed Description Text (16):

At any point in the pretreatment process, desirably prior to passage of the wastewater through the first filtration medium and preferably after passage through a suitable holding tank to remove high density materials, the wastewater can be subjected to any suitable action which reduces the size of particulates in the wastewater, e.g., contact with rotating blades, for the purpose of facilitating the removal of such particulates without unduly adversely affecting subsequent filtration media. The means by which the particulate size is reduced may also be the means by which the wastewater is removed from the holding tank and passed downstream for further processing, e.g., to the strainer and first filtration medium. Thus, for example, the outlet of the holding tank can be connected by way of a suitable fluid pathway to a macerator pump. The macerator pump would draw wastewater out of the holding tank, reduce solid materials to smaller particles, and furnish the pressure to force the wastewater through the remainder of the treatment system. The macerator pump may be positioned outside the holding tank or within the holding tank. If a macerator pump is utilized, it is preferably fitted with an integral inlet screen, which is set into the holding tank, permitting the debris collected on the screen to be backwashed as needed into the bottom of the holding tank for subsequent removal. The inlet screen is preferably sized to reduce the frequency of clean-out while removing all particulates of sufficient size which would otherwise overwhelm the macerator pump.

Detailed Description_Text (20):

At this level of filtration in an efficient and practical graywater treatment system on board ship, it has been found that effective filtration can only be accomplished through the use of dynamic filtration. Specifically, the effective pore sizes of the filter media are so small, and the effective area of the filter media are so limited, that both plugging of the filter media pores and the formation of cake layers adjacent to the surface of the filter media present themselves as problems with the use of ordinary barrier-type filter elements.

Detailed Description Text (21):

For graywater treatment, once-through barrier filtration is not as satisfactory as dynamic filtration for a variety of reasons. Some of these reasons are that the volumes and contaminant concentrations are generally too high for the effective use of once-through filters, an effective backflush system would be prohibitively large, and precoating (which is needed to keep compressible or sticky contaminants away from the filter madium) requires the introduction of additional chemicals which must be eventually disposed of with the concentrated waste.

Detailed Description Text (22):

Dynamic filtration is an extension of the cross-flow filtration concept. The operating principle is to maintain a filter medium free from plugging or fouling by repelling particulate matter from the filter element and by disrupting the formation of cake layers adjacent to the filter medium. These results are accomplished by moving the fluid being filtered fast enough relative to the filtration medium to produce high shear rates as well as high lift forces on the particles, such as by use of rotary, oscillating, reciprocating, or vibratory means. The shear at the fluid-filter medium interface is nearly independent of any crossflow fluid velocity, unlike tangential or crossflow filtration techniques (which suffer from other problems such as premature filter plugging due to compound adsorption and large and nonuniform pressure drops associated with high tangential velocities along the filter length, potentially causing backflow through the filtration medium and reducing filtration).

Detailed Description Text (23):

Dynamic filtration offers a number of performance advantages in the context of the present invention. Very high shear rates can be produced in the dynamic filtration assembly so as to provide enhanced lift to repel small particles and/or to allow high permeate flow rates. Increases in permeate flux rate have been observed to be approximately linear with increased shear rate in some systems. This means that the filter area required can be dramatically reduced over other filtration means. Since shear can be delivered uniformly across the system, uniformly high flux rates can be achieved and maintained across the system such that progressive fouling is eliminated and extended filtration times can be realized. Moreover, high concentrations of agglomerated particulates can be achieved for removal from the treated fluid using dynamic filtration.

Detailed Description Text (24):

A dynamic filtration assembly has the ability to handle a wide range of contaminants, to achieve an appreciably high concentration of retained solids, to be operated continuously over extended periods without the need for filter aids and/or backflushing, and to achieve uniformly high filter performance to minimize the overall system size. The dynamic filtration assembly may be of any suitable configuration and will typically include a housing which contains a filter unit comprising one or more filtration media and a means to effect relative movement between the filtration medium and the wastewater. The filtration media of the filter unit and the means to effect relative movement between the fluid being filtered and the filtration medium may have any of a variety of suitable configurations. A variety of suitable motive means can be utilized to carry out such relative motion, such as, for example, rotational, oscillation, reciprocating, or vibratory means.

Detailed Description Text (25):

The dynamic filtration assembly may be of any suitable device. Suitable cylindrical dynamic filtration systems are described in U.S. Pat. Nos. 3,797,662, 4,066,554, 4,093,552, 4,427,552, 4,900,440, and 4,956,102. Suitable rotating disc dynamic filtration systems are described in U.S. Pat. Nos. 3,997,447 and 5,037,562, as well as in U.S. patent application Ser. No. 07/812,123. Suitable oscillating, reciprocating, or vibratory dynamic filtration assemblies are generally described in U.S. Pat. Nos. 4,872,988, 4,952,317, and 5,014,564. Other dynamic filtration devices are discussed in Murkes, "Fundamentals of Crossflow Filtration," Separation and Purification Methods,

19(1), 1-29 (1990). In addition, many dynamic filtration assemblies are commercially available. For example, suitable dynamic filtration assemblies include Pall BDF-LAB, ASEA Brown Bovery rotary CROT filter, and New Logic V-SEP. While the desired particulate removal can be achieved by any suitable dynamic filtration assembly, it has been f: .nd that the use of vibratory dynamic filtration, such as generally exemplified by the New Logic V-SEP, is particularly well-suited in the context of the present invention.

Detailed Description Text (27):

Any suitable filtration medium can be utilized in the dynamic filtration assembly. Generally, filtration media with finer effective pore ratings are preferred so as to minimize or avoid the need for post-treatment of the wastewater. Thus, while the filtration medium has an effective pore rating of about 5 .mu.m or less, the filtration medium preferably has an effective pore rating of about 1 .mu.m or less, more preferably about 0.5 .mu.m or less. The dynamic filtration medium most preferably has a molecular weight cut-off of about 200 Daltons or less, and, especially desirable, is a dynamic filtration medium which is a reverse osmosis membrane capable of at least 97% salt rejection (which is believed to be equivalent to a membrane having a molecular weight cut-off of about 50 Daltons). The reverse osmosis membrane capable of at least 97% salt rejection is the finest pore rated membrane which is of practical usefulness in the context of the present invention. After passage of the wastewater through the dynamic filtration assembly, the wastewater desirably need not be subjected to further filtration by passage through a filtration medium, but rather can be subjected merely to an adsorbent bed (and possibly other post-filtration treatments such as ozonation and UV exposure) to remove any remaining contaminants.

Detailed Description Text (29):

While the concentrate from the dynamic filtration assembly is ultimately passed by way of a suitable fluid pathway into a suitable concentrate tank, this can be accomplished after the concentrate is recycled for further concentration. Since the dynamic filtration process is improved by higher fluid velocities which tend to reduce the filter medium fouling rate, it is preferred that the dynamic filtration assembly utilize a recirculation pump to take suction from the outlet end of the dynamic filtration assembly and discharge it into the inlet end of the dynamic filtration assembly. Recirculation rates of between one and ten times the outlet flow rate are preferred, with higher recirculation flow rates being most preferred in treating heavily contaminated fluids. Two or more dynamic filtration assemblies can be manifolded in a multiplex system to provide for switch-over to a fresh dynamic filtration assembly when one becomes fouled.

Detailed Description Text (34):

While the filtrate from the dynamic filtration assembly is preferably passed by way of a suitable fluid pathway directly to the adsorbent bed, the filtrate from the dynamic filtration assembly can be passed through a suitable fluid pathway to an ozonation system prior to contacting the wastewater with the adsorbent bed. The ozonation system generates ozone to polish the filtered wastewater inasmuch as ozone is capable of sterilizing wastewater and oxidizing many organic compounds, as well as killing microorganisms and viruses that may have escaped the upstream filters. Such a sanitizing action typically can be effected with an ozone concentration of at least about 0.5 mg/l, preferably at least about 1 mg/l, in the water. The ozone produces no adverse side effects and quickly disappears from the processed water. Ozone can be supplied by any suitable means.

Detailed Description Text (36):

In the case of air feed, compressed air is dried by a dual bed, pressure swing adsorption air dryer. Along with appropriate filters, this unit provides air of the required quality by treating air, preferably compressed air, which passes through a suitable inlet to the pressure swing adsorption air dryer. In the case of oxygen feed, the oxygen in compressed air is concentrated by a purge sweep adsorber. Such a system is similar to that for the pressure swing adsorption air dryer except for the adsorbent and cycle details. Use of an oxygen feed system requires much more air and a larger adsorption system, and, therefore, the air feed system is preferred. The pressure swing adsorption air dryer is used to purify and dry the air to a low dew point, e.g., -70 degree. F. dew point, so that the ozone generator receives filtered, very dry air to perform well and produce high concentrations of ozone over long periods.

Detailed Description Text (45):

The apparatus will preferably further comprise a strainer through which the wastewater is passed prior to passage through the first filtration medium. Such a strainer will

preferably be a <u>screen mesh</u>, as described above in the context of the present inventive method. The first filtration medium preferably has an effective pore rating of about 160 .mu.m or less, and the means for removing free oil preferably comprises means for contacting the wastewater with a material which absorbs free oil, such as cotton (e.g., Turkish towels).

Detailed Description Text (46):

As explained in more detail with respect to the present inventive method, the dynamic filtration assembly is preferably capable of creating shear forces of at least about 20,000 sec.sup.-1, more preferably shear forces of at least about 100,000 sec.sup.-1. The dynamic filtration assembly is most preferably a vibratory dynamic filtration assembly. The second filtration medium preferably has a molecular weight cut-off of about 200 Daltons or less and more preferably is a reverse osmosis membrane capable of at least 97% salt rejection.

Detailed Description Text (49):

In most applications, as described above with respect to the present inventive method, the apparatus will further comprise a holding tank which enables high density material entrained in the wastewater to settle out on the bottom of the holding tank prior to passage of the wastewater through the strainer. The apparatus can also comprise means for reducing the size of particulates in the wastewater prior to passing the wastewater through the first filtration medium. The apparatus will typically further comprise suitable valves (to control the flow of the wastewater), vents (such as a carbon vent filter to deodorize gases vented from the wastewater holding tank and concentrate tank), and the like as are known in the art.

Detailed Description Text (54):
The sample, as received, did not appear to include any large particles. Fifty-five gallons of the graywater was drawn though a stainless steel 24.times.24
--0.014.times.0.014 mesh screen. A household garbage disposal was used to further reduce the size of any large particles. Approximately fifteen gallons of the strained water was filtered using a BDF-LAB Dynamic Microfilter (Pall Corporation). The filter element was a balanced cylinder of Pall S050-3 PSS grade H, S-series porous stainless steel, with a 5 .mu.m absolute rating.

Detailed Description Text (55): Half the outlet flow was concentrate, and half the outlet flow was filtrate. The filter was started using clear water to set operating parameters. The inlet was then switched to graywater, and the operating parameters were adjusted as appropriate. Flow rates were maintained constant through the test by slightly increasing the operating pressure during the course of the test. Although the filter was tested for only a short time, it was evident that the filter did not foul as quickly as a standard filter. Apparent turbidity in the filtrate was significantly lower than that of the influent, while turbidity of the concentrate was higher than that of the influent. The permeate flow rates and annulus pressures at 10 and 30 minutes into the test are set forth below.

Detailed Description Text (63):

A fifty-five gallon drum of graywater was drawn from the David Taylor Research Center and filtered through a series of filters characterized by decreasing pore size. The first filter was a knit mesh screen which simulated a strainer. The second, third, and fourth filters simulated the filtration of the dynamic filtration assembly. The second filter was an Ultipor.RTM. GF 3 .mu.m absolute filter (Pall Corporation), while the third filter was a Sanitary Filter with a nylon membrane and a 0.04 .mu.m absolute rating (Pall Corporation), and the fourth filter was an Ultrafilter.RTM. model VIP-3017 (Asahi), with a cut-off molecular weight of 6000 Daltons.

Detailed Description Text (64):

The fourth filter was rinsed with 3 .mu.m absolute deionized water for approximately 20 hours and then drained prior to testing. The drum of graywater was pressurized to approximately 5 psig with air, which forced the graywater through the filters at a total flow rate of less than 0.5 gpm. The concentrate flow rate from the fourth filter was approximately one-fourth that of the filtrate (purified water) flow rate. Samples of the filtrate were taken at three different locations on the test system: (i) downstream of the first filter, (ii) downstream of the second and third filters, and (iii) downstream from the fourth filter. The filtrate was allowed to flow from the first two sample taps for 30 seconds before the samples were taken, and the fourth filter was flushed with process fluid for ten minutes prior to collecting samples.

Detailed Description Text (65):

The filtration system definitely improved the clarity of the graywater, and the odor and foaming of the graywater were also reduced. The flow rate gradually dropped through the test while a constant pressure was maintained in the drum. Examination of the system indicated that either the second or third filter, or both, were becoming plugged. The use of an actual dynamic filtration assembly will preclude such plugging. Examination of the concentrate in the housing of the second <u>filter</u> revealed that it was much darker than the original graywater fluid. The test results are set forth below.

Detailed Description Text (66):

The test results indicate that the combination of the various filters can meet total suspended solids and fecal coliform requirements, while the addition of acid feed may be required to correct the strongly alkaline condition. The use of a coagulant upstream would enhance the filtration effect achieved by the dynamic filtration. The test results also indicate that ozone and UV light irradiation would be useful to further reduce the BOD level.

Detailed Description Text (69):

Using a barrel pump, the graywater was passed through a 30.times.30.times.0.0065 screen (wires/linear inch.times.wires/linear inch.times.wire diameter (inches)) into pails to remove large debris. The removed debris consisted of hairs, thick stringy material, flakes, and other soft media. The bottom 2-3 inches of each graywater drum contained a thick, black oily substance.

Detailed Description Text (70):

The graywater in the pails was then poured through a 160 .mu.m metal screen filter. The thus filtered graywater was opaque, ink black and contained no trace of soap or soap suds. A strong, pungent odor was emitted by the freshly poured graywater, but subsided within a few hours.

Detailed Description Text (71):

The graywater was then subjected to dynamic filtration utilizing the vibratory dynamic filtration assembly PALL-SEP VMF Series L (Pall Corporation) and one of two filtration media, either a 200 molecular weight cutoff nanofiltration membrane (with a 80% minimum salt rejection rating) or a 97% salt rejection rated reverse osmosis membrane. The operating conditions of the dynamic filtration assembly were as follows:

Detailed Description Text (72):

Utilizing the nanofiltration membrane, approximately 80 gallons of graywater were treated over a three-week period. At the end of this period, 99.31% of the fluid had been removed from the system as permeate. The total permeate collected, i.e., 287.53 l , indicated a loss of 13.3 l or 3.52 gal of graywater. The loss in graywater was attributed to the evaporation of volatile organic compounds which were noticeably emitted and water vapor. The total graywater quantities recorded were reduced by 4.4% to account for the evaporative loss. As the % permeate/total graywater progressed toward 99.31%, the permeate flowrate decreased from about 73 ml/min to about 47 ml/min.

Detailed Description Text (73):

Utilizing the reverse osmosis membrane, approximately 72 l of graywater were treated with a permeate recovery of about 68 l , or about 94.31%. As the % permeate/total graywater progressed from about 89% to about 94%, the permeate flowrate decreased from about 24.4 ml/min to about 21.3 ml/min.

Detailed Description Text (75):

The permeate derived from the nanofiltration membrane exhibited a mild, pungent odor and had a very slight yellow tint. The permeate derived from the reverse osmosis membrane was crystal clear and did not emit any odor. Both permeates met U.S. requirements for discharge into open water; however, the permeate derived from the reverse osmosis membrane was generally more free of contaminants than the permeate derived from the nanofiltration membrane. The permeate derived from the nanofiltration membrane would preferably be further processed by contacting the permeate with a suitable adsorbent bed to ensure compliance with applicable discharge laws, while the permeate derived from the reverse osmosis membrane could possibly avoid the need for such further treatment.

Detailed Description Paragraph Table (4):

first <u>filter</u> second + third <u>filters</u> fourth TS (mg/l) 1100 980 580 TSS (mg/l) 26 2 1 BOD (mg/l) 220 74 47 COD (mg/l) 540 210 120 O&G (mg/l) 110 9 1 FC (#/100 ml) 19 <1 <1

ph 10.1 10.1 5.5	pH 10.1	10.1	9.9	
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Detailed Description Paragraph Table (5):

Nanofiltration Reverse Osmosis Membrane Membrane graywater feed 200 psig .+-. 5 psig 200 psig .+-. 1 psig pressure graywater feed 72.degree. F. .+-. 1.degree. F. 72.degree. F. .+-0.3.degree. F. temperature graywater return 0.40 gpm .+-. 0.01 gpm 0.40 gpm .+-. 0.03 gpm flow rate PALL-SEP VMF 1.25 in 1.25 in oscillatory displacement PALL-SEP VMF 63.25 cps .+-. 0.10 cps 63.25 cps .+-. 0.05 cps oscillatory frequency

Detailed Description Paragraph Table (6):

Graywater Graywater After After Graywater Dynamic Dynamic Before Filtration Filtration Dynamic (Nanofiltration (Reverse Osmosis Total Solids Filtration Membrane) Membrane) 14,600 {683} [15] (mg/l) Total Suspend- 85.8 [1.5] [2] ed Solids (mg/l) Biochemical ND (<60) [11] [8.8] Oxygen Demand (mg/l) Chemical 111 40.2 ND (<2) Oxygen Demand (mg/l) Oils & Greases 33.1 1.7 2.7 (mg/l) Fecal ND (<4) NT NT Coliforms (#/100 ml) pH 8.55 ND: none detected NT: not tested []: probably background level samples collected in nonsterilized plasti containers { }: dubious result (actual value likely lower)

Other Reference Publication (2):

Culkin, "Vibratory Shear Enhanced Processing: An Answer to Membrane Fouling?," Chemical Processing (Jan. 1991).

Other Reference Publication (3):

Culkin et al., "New Separation System Extends The Use of Membranes," Filtration & Separation, 29(5) (Sep./Oct. 1992).

CLAIMS:

- 3. The method of claim 2, wherein said strainer is a screen mesh comprising about 10-50 wires por linear inch in each direction with such wires having a diameter of about 0.002-0.02 inch.
- 11. The method of claim 9, wherein said second filtration medium is a reverse osmosis membrane capable of at least 97% salt rejection.
- 37. The apparatus of claim 36, wherein said strainer is a screen mesh comprising about 10-50 wires per linear inch in each direction with such wires having a diameter of about 0.002-0.02 inch.
- 45. The apparatus of claim 44, wherein said second filtration medium is a reverse osmosis membrane capable of at least 97% salt rejection.

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L10: Entry 6 of 17

File: USPT

Jan 21, 1997

DOCUMENT-IDENTIFIER: US 5595349 A

TITLE: Continuous flow rotary materials processing apparatus

Abstract Text (1):

A continuous flow, rotary processing apparatus for horizontal transport of material at a predetermined rate. Material from an infeed conveyor enters a continuous system including at least one rotary drum operating about a horizontal axis. Each drum has a perforated circumferential wall with lateral lifters to induce flow and tumbling motion to the enclosed material. A flow control mechanism at the discharge end of each drum controls residence time of the material being processed. Material is fed and discharges from each drum through axial openings in which the flow control devices are mounted.

Brief Summary Text (11):

Next, de-watered material enters a rotating evaporative dryer section of the system where hot air flows over the cleaned material to remove any remaining moisture. Preferably, most of the heated air is recirculated to conserve energy. The dryer drum, like the proceeding drums, is fabricated of screen to enable air passage through the drum and over the moist plastic.

Drawing Description Text (16):

FIG. 18 is a side view of the particle filter components of the wash water recirculation system.

Detailed Description Text (6):

The washer section 14 basically comprises a housing 40, a washer drum 41, a drive mechanism, a plurality of sprayers 51, and an egress control chute 43. The housing 40 is shown to be a stationary, generally square enclosure with top, bottom, side and end walls. The housing 40 is shown to be supported by a frame structure 25 which also elevates the apparatus 10 off of the ground. The drum 41 is a cylindrical enclosure or chamber, the central axis of which is aligned horizontally between the front and back end walls. The diameter of the drum 41 is such that it encompasses the majority of the housing 40 interior volume to maximize processing space, but still allows room for the remaining elements of the section 14, as further described below. The drum 41 volume is dependent upon the infeed rate of material and the speed of the drum 41. The length of the drum 41 extends substantially from end to end of the housing 40. The drum 41 is preferably constructed of a metal mesh or screen material of the pore size such that it allows wash fluids to penetrate, but also prevents processed material particles from escaping its enclosure. As best shown in FIG. 7, the drum 41 is supported by a pair of rollers 46, which are fixed to the housing 40. The drum 41 has six (6) vanes 45 disposed on the interior of its circumferential wall at 60 degree intervals and aligned longitudinally with respect to its center axis. The vanes 45 function to lift and impart vertical motion to material inside drum 41 during rotation. The number of vanes 45 and their dimensions are variable to influence the rate of flow and residence time of the material in the apparatus 10, and further depend upon several factors, including the nature and size of the material being processed, the velocity of drum 41 rotation and the structure and orientation of the egress control chute 43. Generally, the greater the surface area of the vanes 45, the greater the upward lift they provide, which increases flow rate as described further below.

Detailed Description Text (8):

Referring to FIGS. 15-17, the sprayers 51 are shown to be disposed above and outside the washer drum 41, and connected to a wash water conduit 50. The sprayers 51 preferably emit a constant, high volume stream of heated water, or a suitable cleaning agent or solvent through the drum mesh to saturate the tumbling material therein. The expected path of the material is designated 66 and 67 in FIGS. 15 and 16. Fluids mixed with contaminates extracted from the material migrate to the bottom of the apparatus 10 where they are directed by a wash fluid collector 42 to a wash water recirculation system components 27 and 28, which are described further below.

Detailed Description Text (14):
Referring to FIGS. 17-23, the wash water recycling system receives fluids drained the washer and dewater sections 14 and 15, which are contaminated with wastes extracted from the processed material. The system then removes both large and relatively small contaminant particles from the fluid. The system preferably heats the recycled fluid and then returns the fluid to the sprayer heads 51 in the washer section 14 via supply conduit 50.